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March 29, 2006

Mr. Richard Ho
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U. S. Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
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Subject:

Submittal of Final Vapor Intrusion Evaluation Work Plan for

Operable Unit 1 of the Quanta Resources Corporation Superfund Site

Dear Mr. Ho:

On behalf of Honeywell International, Inc. and the Edgewater Site Administrative Group (ESAG), CH2M HILL is pleased to present seven copies of the Final Vapor Intrusion Evaluation Work Plan for Operable Unit (OU) 1 of the Quanta Resources Corporation Superfund Site in Edgewater, New Jersey. This document has been prepared in accordance with the requirements of the Administrative Order on Consent (AOC) II-CERCLA-2003-2012 and the USEPA approval letter dated March 20, 2006. Attached to this letter are responses to all comments on the work plan submitted by the regulatory agencies.

One copy of the document has been provided to Clay Monroe at the USEPA Office of Regional Counsel, and four copies of the document have been provided to Robert Hayton of the New Jersey Department of Environmental Protection.

If you have any questions about these draft submittals, please contact me at 973-316-0159 x4505 or Tim Metcalf of Honeywell at 973-455-4107.

Sincerely,

CH2M HILL

R. Scott Stoldt, CPG Project Manager

Cc:

Clay Monroe/USEPA Region 2
Robert Hayton/NJDEP
Tim Metcalf/Honeywell

**Edgewater Site Administrative Group** 

# CH2M HILL Responses to Comments from USEPA and NJDEP: Quanta Resources Superfund Site Vapor Intrusion Evaluation Work Plan

Responses to Comments from USEPA Region 2, February 13, 2006

Comment 1: Page 2-3: In the third paragraph on this page, please correct the spelling of "fluorene."

Response: corrected.

Comment 2: Page 3-1: Section 3.1 suggests that only BTEX and naphthalene will be analyzed. EPA Region 2 typically analyzes for all contaminants on the TO-15/17 list. Please clarify that all contaminants will be included in the analysis of subslab soil gas and indoor air.

Response: The text has been revised in Section 3.1to reflect analysis of all constituents on the TO-15 target analyte list (we do not propose to use Method TO-17 as discussed below).

Please note that Section 3.1 refers to the target analyte list presented in the vapor investigation Quality Assurance Project Plan (QAPP). That target analyte list is based on the USEPA Region 2 model vapor intrusion workplan and QAPP guidance documents. The tables displaying the complete target analyte list are presented both in the workplan (Table 4) and QAPP amendment ( (Tables A-1 and A-3) .

There are a few constituents that both cannot be feasibly analyzed in air samples and are unlikely to be constituents in coal tar or waste oil materials associated with the Quanta Resources Site. These are:

- Acetylene: this constituent is not considered to trap well in the TO-15 method, and therefore has poor instrument response during analysis. In addition, a preliminary evaluation of potential human health risks associated with this substance indicates it is simply an asphyxiant and potential fire/explosion hazard, at percent-level in air. There are investigative methods other than SUMMA canister sampling and TO-15 analysis that would be more useful to evaluate the potential risks associated with this chemical, if necessary.
- Bromochloromethane: this is the internal standard used in the TO-15 method (see Section 92.2.3 of USEPA's method description, included as Attachment 1 to the QAPP). Therefore, it cannot be analyzed as a constituent in environmental samples.
- Chloroprene (2-chloro-1,3-butadiene): this will be reported as a tentatively identified compound, and evaluated further if needed. Chloroprene is synthesized from butadiene gas, and is used in rubber manufacturing; it is unlikely to be a constituent in coal tar or waste oil materials associated with the Ouanta Resources Site.

• Ethyl acrylate: this will be reported as a tentatively identified compound, and evaluated further if needed. Ethyl acrylate is used in manufacturing polymers, and is synthesized from precursors including acetylene, ethyl alcohol, chlorohydrin or acrylonitrile; it is unlikely to be a constituent in coal tar or waste oil materials associated with the Quanta Resources Site.

We do not propose to use Method TO-17 for this initial sampling event. Method TO-17 involves the use of sampling pumps and sorbent tubes which would significantly increase the complexity of field and laboratory activities relative to the amount of usable information produced from this sampling method. We observe that EPA's model work plan and QAPP for vapor intrusion does not refer to Method TO-17.

Comment 3: Page 3-2: This page presents four response levels, based on a comparison of indoor air data to appropriate criteria. However, EPA Region 2 recommends collecting subslab soil gas initially and evaluating the need to collect indoor air data. Often, decisions can be made using only the subslab data. Therefore, it is difficult to determine how the response levels will be used, when EPA Region 2 is not necessarily in agreement with the data that will be collected.

Response: Based on our understanding of site conditions and concern for the well-being of building occupants at 115 River Road, we believe that the building investigation warrants a Tier 3 – Site-Specific Pathway Assessment, consistent with USEPA's draft vapor intrusion guidance, which states:

"... this guidance recommends: (1) direct measurement of foundation air concentrations before any indoor air measurements; (2) direct measurement of indoor air concentrations coupled with a home survey (see Appendix H) and sampling to identify background sources of vapor in ambient (outdoor) and/or indoor air; 3) removal of all indoor air sources before sampling indoors; and (4) complementary site-specific mathematical modeling as appropriate. The sampling of foundation air (e.g., subslab and /or crawlspace air) and ambient (outdoor) air in conjunction with indoor air is intended to distinguish the exposures that originate from subsurface contaminant vapor intrusion from those due to background sources." (page 38)

With respect to seasonal variability of vapor concentrations, we propose conducting indoor air sampling during winter conditions while the heating system is in operation with minimal air exchange rate. This is consistent with air sampling approaches referenced in USEPA's draft vapor intrusion guidance. We are concerned that if the investigation approach is sequenced, there is considerable risk of missing the opportunity in early 2006 to collect indoor air samples under winter conditions.

In addition, we acknowledge that subslab vapor analytical data is used in conjunction with outdoor and indoor air data to distinguish background from potential vapor intrusion. One of the uses for the subslab data is to help identify constituents of interest for indoor air sampling. Given the timing of the investigation activities relative to seasonal conditions, we are prepared to analyze the initial round of air samples for the full target analyte list, and use the subslab

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and outdoor air sampling results to refine the list of constituents of interest for additional indoor air sampling events, if warranted.

In reviewing the decision flow chart for using subslab data under Question 6 in USEPA's draft vapor intrusion guidance, we acknowledge that subslab data can be used either to exit the vapor intrusion pathway identification process (see Question Q6(f)), or collect additional information including indoor air sampling – these represent the decisions that are based on subslab data, according to USEPA's 2002 draft vapor intrusion guidance. Moreover, this approach is also consistent with USEPA's vapor intrusion 2003 training (see the presentation, *An Overview of Question 6 of the Guidance – Site-Specific Assessment*, by Dominic DiGiulio).

We also note that recent regulatory vapor intrusion guidance (NYSDOH, 2005) <sup>1</sup> recommends collecting indoor air and subslab samples concurrently.

Given the seasonal constraints for investigation (capturing an indoor air sampling event under winter conditions), we believe that collection of subslab and indoor air samples concurrently is needed to be able to promptly evaluate the potential vapor intrusion pathway. Based on a careful reading of USEPA's 2002 draft vapor intrusion guidance, we do not feel that collecting indoor air and subslab sampling in this manner is in conflict with the intent of USEPA's screening process.

USEPA Region 2's vapor instrusion decision matrix (as discussed during the February 17, 2006 teleconference has been incorporated into the vapor intrusion evaluation work plan (see Tables 3a and 3b).

Comment 4: Page 4-1: This section begins with a building survey utilizing industrial hygiene smoke tube techniques. EPA Region 2 suggests beginning with collecting subslab soil gas data to determine whether or not vapors are actually collecting under the building. If vapors are not detected at concentrations of concern or at concentrations that suggest that this phenomenon is occurring, then there is no need to use the industrial hygiene smoke tube techniques.

Response: The building survey is necessary prior to conducting subslab sampling in order to identify proposed sampling locations, coordinate access to those locations with the building owners/managers, and to perform utility clearances prior to installation of the subslab sampling probes. Prior to conducting subslab sampling, the building survey will be conducted during which the proposed subslab sampling locations will be identified. The proposed sampling locations will then be presented to USEPA Region 2 for review and approval. As discussed in the work plan schedule, the expectation is for a prompt review and resolution on the subslab sampling locations so that sampling can commence during the current winter season.

Recent regulatory guidance for vapor intrusion investigations) recommends the use of smoke tubes or other devices, in conjunction with subslab sampling, to evaluate pressure relationships and air flow patterns, especially between floor levels and

<sup>&</sup>lt;sup>1</sup>New York State Department of Health. 2005. Guidance for Evaluating Soil Vapor Intrusion in the State of New York, Draft February 2005.

between suspected contaminant sources and other areas (NYSDOH, 2005). We recommend that these tools be employed to refine the conceptual site model of the potential vapor intrusion pathway, and for identifying proposed locations for indoor air samples. However, use of smoke tubes, if deemed necessary, will be minimized.

Comment 5: Page 4-2: Section 4.2 states that shallow soil gas sampling will be conducted to further refine the preliminary conceptual site model. EPA Region 2 does not recommend collecting shallow soil gas samples. First, with the existence of a building, subslab soil gas is recommended to evaluate the potential for vapors to migrate from a subsurface source to underneath the building. Second, shallow soil gas is likely to be impacted by climatological conditions, such as rain or snowmelt, which will displace vapors from the pore space, or wind, which will likely dilute any vapors that migrate to the shallow soils.

Response: Although we believe that shallow soil gas sampling would be beneficial to the understanding of potential vapors where buildings do not exist at the Site, shallow soil gas sampling will not be collected as part of the vapor intrusion evaluation for the 115 River Road building as requested by USEPA Region 2. Section 4.2 has been deleted from the workplan.

Comment 6: Page 4-3: The second bullet on this page states that an attenuation factor of 0.01 will be used. EPA Region 2 uses an attenuation factor of 0.1 to account for subslab to indoor air migration. EPA Region 2 has developed a process to assess subslab and indoor air concentrations relative to the acceptable risk range and noncancer hazard quotient that incorporates a factor of 0.1 when evaluating indoor air and subslab soil gas.

Response: Text has been modified to reflect the application of an attenuation factor of 0.1 as requested by USEPA during our February 17, 2006 telephone teleconference (see Section 3.2). Although USEPA's 2002 draft vapor intrusion guidance specifies the use of the attenuation factor of 0.1 for developing subslab screening values, USEPA received significant comments on the derivation of the default attenuation factor values, and in response has sponsored an extensive review of the empirical database of vapor intrusion investigation results. The results from that review were presented in 2005<sup>2</sup>, and indicated that a subslab-to-indoor air attenuation factor of 0.01 to 0.02 is probably more appropriate for non-degrading chlorinated volatile organic compounds. These attenuation factors are probably conservative for many hydrocarbon constituents, based on a review of the literature presented in New Jersey's recent vapor intrusion investigation guidance document (NJDEP, 2005)<sup>3</sup>.

As discussed with USEPA Region 2 during the February 17th teleconference, a subslab-to-indoor air attenuation factor of 0.1 will be used initially to calculate subslab screening levels for this vapor intrusion evaluation. The results from this initial sampling event can then be used to develop building-specific attenuation factors and develop subslab screening levels more appropriate for site conditions.

<sup>&</sup>lt;sup>2</sup> Hers, I., H. Dawson and R. Truesdale. 2005. *Revising the Empirical Attenuation Factors: Data Analysis and Preliminary Results*. Presented at the Vapor Intrusion Workshop, 16th Annual West Coast Conference on Soils, Sediments and Water, San Diego, California. March 14, 2005.

<sup>&</sup>lt;sup>3</sup> New Jersey Department of Environmental Protection. 2005. New Jersey Vapor Intrusion Guidance, October 2005.

Comment 7: Page 4-3: The third bullet states that when subslab concentrations exceed their screening values, further evaluation will be necessary. Please note that when subslab concentrations exceed screening values, it may be appropriate to pursue some type of remediation, rather than continue sampling.

Response: We acknowledge that USEPA's 2002 draft vapor intrusion guidance recommends further evaluation in the case of subslab concentrations exceeding their screening values. In addition, a review of recently published regulatory vapor intrusion guidance documents (NYSDOH, 2005; NJDEP, 2005) generally recommends using a combination of indoor air and subslab data to make mitigation decisions. We draw USEPA Region 2's attention to Table 4 in the workplan and Table A-2 in the QAPP amendment, which shows that the subslab screening levels correspond to a target excess lifetime cancer risk level of 1 x 10-6 and a noncancer hazard quotient of 0.1. Detecting subslab concentrations based on these screening levels will facilitate additional indoor air sampling for confirmation and characterization purposes. Based on the magnitude of subslab concentrations and indoor air concentrations, mitigation, as appropriate, will be evaluated. As discussed in the response to Comment 3, USEPA's vapor intrusion decision matrix (now included as Tables 3a and 3b) will be evaluated to identify further steps (either investigation or mitigation) based on initial investigation results.

Comment 8: Page 4-3: The fourth bullet states that when concentrations in the subslab are below levels of concern, no additional evaluation will be necessary. Please note that this may not always be the case. For example, when subslab concentrations fall within the risk range, this suggests that the potential for accumulation beneath the slab exists. Further evaluation may be necessary to determine if the accumulation may increase, if the source has been identified or remediated, or if the potential for these vapors to migrate to indoor air exists.

Response: The subslab probes will be installed so that they can remain in place longer-term, in case additional sampling events are needed.

Comment 9: Page 4-3: Section 4.4: Typically, EPA Region 2 collects all ambient samples concurrent with indoor air samples. The text suggests that only those samples collected from the roof of the former Spencer Kellogg building will be collected concurrent with indoor air samples.

Response: All ambient samples will be will be collected concurrently with the indoor air samples, as noted in new Section 4.3.

Comment 10: Page 5-2: Section 5.5: EPA Region 2 typically collects subslab data analyzed with quick turnaround times to determine if vapors are collecting under buildings. If this condition is found to exist, then indoor air and subslab are collected and analyzed with complete QA/QC validation to ensure results of sufficient quality.

Response: Please see the response to Comment 3. We believe that collecting indoor air and subslab samples concurrently will provide a more expedient approach to evaluating the potential vapor intrusion pathway.

Comment 11: Table 3: All TO-15/17 constituents should be included. Also, EPA Region 2 has developed an approach to identify levels of indoor air and subslab soil gas

concentrations that can be used to determine future activities. Finally, the table states that the concentration provided as screening levels are for indoor air. As previously stated, EPA Region 2 typically collects subslab soil gas to determine if vapors are actually collecting beneath a building prior to collecting indoor air samples. This table does not reflect the current paradigm used by the Region.

Response: Please see the response to Comment 2 regarding the constituents to be included for investigation. The proposed target analyte list reflects USEPA Region 2's model work plan and QAPP for vapor intrusion investigations.

The screening levels shown in Table 4 incorporate USEPA Region 2's approach to identifying screening levels in indoor air. Please see the response to Comment 6 regarding the development of subslab screening levels.

Please see the response to Comment 3 regarding the sequencing of subslab and indoor air sampling. We believe that collecting indoor air and subslab samples concurrently will provide a more prompt response in evaluating the potential vapor intrusion pathway.

Comment 12: Table 4: Please explain why ATSDR MRLs are used. Also, why is an MRL provided for benzene, a carcinogen?

Response: The response levels in Table 3 (not Table 4) are intended to provide activities that appropriately address a range of possible indoor air concentrations. The MRL values are intended to identify possible conditions warranting prompt action to reduce potential exposures, and identify specific activities to be undertaken for exposure reduction. Development of such response levels that cover a range of possible situations is important at the planning stage of a vapor intrusion investigation to be able to provide prompt responses.

The acute MRL is not intended for use as a long-term exposure value; as can be seen from Table 3, detecting concentrations at this level in indoor air would trigger prompt activities to reduce potential exposures. However, indoor air screening levels will be reevaluated as discussed during the February 17th teleconference. As discussed in the response to Comment 3, USEPA Region 2 will provide its vapor intrusion decision matrix. This matrix (presented in Tables 3a and 3b in the revised workplan) has been used to revise the response levels (now shown in Table 4 of the revised workplan) and link these to further steps (either investigation or mitigation) based on initial investigation results. Note that the workplan also includes NJDEP Rapid Action Levels and Health Department Notification Levels, as requested by NJDEP.

Comment 13: Table 5: This table requires revision to be consistent with the EPA Region 2 approach for assessing potential indoor air impacts. The purpose of collecting soil gas needs to be clearly defined, as it does not appear to have utility in this exercise.

Response: Comment noted. Section 4.2 discussing soil gas sampling has been removed from the workplan. Resolution of previous comments should address USEPA Region 2's concerns regarding the sampling approach presented in this table. Please see the response to Comment 5 regarding soil gas sampling.

#### Responses to Comments from NJDEP, February 17, 2006

Comment 1: Pre-Sampling Investigation: The information on Appendix B of the Oct 2005 NJDEP Vapor Intrusion Guidance Document (VIG) must be included in the building survey questionnaire contained in the QAPP amendment attachment 3.

Response: this information will be captured in the building survey questionnaire. NJDEP's questionnaire has been included in Attachment 3 to provide assurance that the needed information is captured during building survey activities.

#### Comment 2: Section 4.2: Soil Gas sampling:

Shallow soil gas sampling is proposed along the northern side of the Former Spencer Kellogg building. Eight soil gas samples are proposed along two roughly east-west transects between the Former Spencer Kellogg building and the Quanta Resources site (see Figure 1). The sample locations will be spaced approximately 200 feet apart along the length of the building (excluding the pier construction within the Hudson River) and at two distances from the building foundation. Four samples are proposed to be collected from each transect. At each transect, one boring will be placed along the southern border of the Quanta site (approximately 60 feet north of the Former Spencer Kellogg building). The second trench (borings) will be collected close to the building foundation considering limitations due to obstructions and subsurface utilities (generally within 15 feet of the building).

a. It is stated in Section 5.2 on page 5-1, that soil gas grab samples will be collected from the probes into evacuated 6-liter stainless steel SUMMA canisters. This is unacceptable. The NJDEP requires that soil gas samples be collected in 1-liter containers to avoid short-circuiting or dilution of the sample with atmospheric air. This section must be consistent with Section 4.3.3 of the QAAP.

Response: As discussed in the response to USEPA Region 2's Comment 5, soil gas sampling will not be conducted at this time, as part of the 115 River Road vapor intrusion evaluation.

#### Comment 3: 4.3 Synoptic Sub-slab and Indoor Air Sampling

Following the building walkthrough survey, sub-slab and indoor air samples will be collected from the Former Spencer Kellogg building. The number and locations for the sub-slab indoor air samples will be identified following completion of the walkthrough survey. The consultant proposes to collect sub-slab and indoor air samples synoptically.

a. The following sub-slab conditions must be determined prior to drilling into the sub-slab: (depth to groundwater, underground utilities and presence of a vapor barrier)

Response: These conditions will be identified during the building walkthrough survey.

b. It is stated on page 4-3, that the results from the indoor air samples will be evaluated by comparison to the response levels shown in Table 3. This is not acceptable. NJDEP requires that the indoor air analytical results be compared to the more conservative of the USEPA criteria and the NJDEP Indoor Air Screening Levels (IASLs), Rapid Action Levels (RALs) and Health Department Notification Levels (HDNLs) found on Tables 1 and 2 of the Oct 2005 Vapor Intrusion Guidance Document (VIG). The sub-slab soil gas results

should be compared to the NJDEP Soil Gas Screening Levels (SGSL) found in Table 1 of the VIG.

Response: The work plan text will be revised to include these screening levels. The revised levels are presented in Tables 4 and 5.

#### Comment 4: Section 4.4 Ambient (Outdoor) Air Sampling

Ambient air samples are proposed from four outdoor locations beyond the footprint of the Quanta site. These samples are proposed from unspecified locations beyond the extent of dissolved phase plumes or non-aqueous phase liquid in groundwater. It is stated that two of the samples will be collected in the predominantly upwind direction from the site while two samples will be collected in the predominantly downwind direction from the site. The locations of the samples have not been determined. Two ambient air samples are also proposed from the roof of the Former Spencer Kellogg building, near outdoor air intake vents. These two samples are proposed to be collected during the synoptic indoor air and sub-slab sampling event. It appears that there will be a total of 4 or 6 ambient air samples. Clarification is required. Also, NJDEP requests that all ambient air samples be collected concurrent with the indoor air samples, not just the two rooftop samples.

Response: the approach for collection of ambient outdoor air samples has been revised based on further comments from USEPA Region 2 (see below).

#### Comment 5: Section 5.4, Air Sampling (Indoor and Outdoor)

a. It is stated on page 5-2 that pressure and temperature will be obtained from the National Weather Service. NJDEP requires that ambient pressure and temperature readings also be collected inside the building using portable meteorological instrumentation. Temperature and pressure readings are required for each sample location. This information must be added to the SOPs in Attachment 2 and the sample data sheets in Appendix C.

Response: The work plan text has been revised to include indoor barometric pressure and temperature measurements to be collected using portable instruments as requested. See Section 5.2 for the revised text.

b. It should be noted in this section that indoor air samples are required from different floors within the building. NJDEP recommends, at a minimum, that samples be collected from locations on the lowest floor (basement) and the level immediately above it. Also note that ambient and indoor air samples are required to be collected at approximate breathing zone height (3'-5'). Basement samples must be positioned as close as possible to any potential source areas (sumps, foundation cracks, etc.)

Response: Proposed indoor air sampling locations, identified following the walkthrough survey, as shown in Table 6 of the revised workplan.

#### Comment 6: Section 7, Data Evaluation, Interpretation and Reporting:

#### a. TABLE 3, Possible Response Levels in Indoor Air

The work plan indicates that the data from the indoor air samples will be evaluated by comparison to the response levels shown in Table 3. This is not acceptable. NJDEP

requires that the indoor air analytical results be compared to the more conservative of the USEPA PRG criteria and the NJDEP Indoor Air Screening Levels (IASLs), Rapid Action Levels (RALs) and Health Department Notification Levels (HDNLs) found on Tables 1 and 2 of the Oct 2005 Vapor Intrusion Guidance Document (VIG). The sub-slab soil gas results should be compared to the NJDEP Soil Gas Screening Levels (SGSL) found in Table 1 of the VIG.

Response: see the response to NJDEP comment 3a for discussion of revisions of the response levels.

Note that USEPA Region 2 has requested that subslab screening levels be calculated using an attenuation factor of 0.1. This will provide more conservative levels than presented in NJDEP's vapor intrusion guidance.

b. NJDEP requires that the response levels presented in Table 3 be re-evaluated. It is required that the lowest of the NJDEP IASLs and the USEPA PRGs be used for the most conservative screening levels (Levels 1 and 2) in this table. Note that the NJDEP IASL for Toluene is 420 ug/m³, which is lower than the 520 ug/m³ presented in the table.

Response: The work plan text has been modified as stated above. See also our response to Comment 6a.

c. Note that NJDEP requires that if the indoor air sample results exceed the RALs, confirmatory samples must be collected immediately upon receipt of the data. Note that the NJDEP RALs for Benzene and Toluene are lower than the Level 2 Screening criteria in Table 3. This must be changed. All of the Level 3 and 4 Screening Criteria presented in Table 3 are consistently higher than the NJDEP Health Department Notification Levels (HDNLs) presented in the VIG. This is also unacceptable and must be changed. It is required that the Possible Response Levels and recommended actions in Table 3 be consistent with the most stringent of USEPA criteria and the NJDEP criteria and recommended responses presented in section 7.8 of the VIG. Response levels are only presented for BTEX and Naphthalene. Other VOCs may be detected at levels of concern. The Table should reference the NJDEP IASLs and other USEPA screening levels for other potential contaminants of concern at the site.

Response: Additional clean and certified canisters will be available to be able to quickly collect confirmation samples should these be needed. As discussed in the response to Comment 6a, the levels have been revised to incorporate consideration of NJDEP guidance. Note that some of the constituents of interest (BTEX and naphthalene) are also normally present in ambient air. As discussed in NJDEP's vapor intrusion guidance, background concentrations will be considered when interpreting indoor air sampling results. We acknowledge that NJDEP's guidance states: "[b]ackground contaminant levels, particularly ambient air results, supercede the Table 1 values when higher since the Department does not require remediation to levels below background concentrations" (page 131). Screening levels have been developed for all of the target analytes detected by Method TO-15. These were included in Table 4 of the workplan and Table A-1 of the amended QAPP.

Response to Comments from USEPA Region 2 on Quality Assurance Project Plan Amendment, received March 1, 2006

#### Comment 1: General

a. Region 2 has recently adopted the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), June 2005, as the standard format for preparing QAPPs for all Superfund, RCRA, and Brownfields projects within the Region. It should be noted that QAPPs previously prepared do not have to follow the UFP guidance, however, any site-specific QAPPs, a significantly revised generic QAPP, or any new QAPPs prepared for sites in Region 2 will need to follow this new format. The reference pertaining to the UFP-QAPP is located at: <a href="http://www.epa.gov/fedfac/documents/qualityassurance.htm">http://www.epa.gov/fedfac/documents/qualityassurance.htm</a>.

Response: the QAPP prepared for this workplan is an amendment to the existing OU1 QAPP, submitted in October 2005. This QAPP amendment adds methods and quality assurance procedures for air and subslab sampling using evacuated stainless steel canisters and analysis using EPA Method TO-15. We expect that the procedures described in the QAPP Amendment (Appendix A of the Work Plan) will provide data of comparable quality and documentation consistent with the UFP-QAPP.

b. The first page of the Work Plan/QAPP should contain the signature and printed name/organization/date of the: investigative organization's project manager; investigative organization's project QA officer; lead organization's project manager as well as other appropriate personnel.

Response: The QAPP amendment includes a signature page prepared in the same manner as for the OU1 QAPP.

c. The Work Plan/QAPP should use a document control format to identify the current version of the document and to ensure that only that version is being used by all participants.

Response: version numbers will be added if any changes are made to the QAPP amendment. Note that the format is consistent with the OU1 QAPP.

#### Comment 2: Work Plan

a. Page 4-2 - A definition of the term "synoptically" should be provided.

Response: a footnote has been added defining the term as meaning indoor, outdoor and subslab samples will be collected simultaneously so as to characterize a snapshot of conditions.

b. Section 4.3, Page 4-3 - A reference to the screening levels to be used for the subslab sampling should be included in this section.

Response: the response levels are addressed in Section 3.2 of the workplan.

c. Section 5.5, Page 5-2 - Remove references to "CLP Level" since they are no longer used. Use CLP-like deliverables instead.

Response: this language has been removed.

d. Section 7, Page 7-1 - It is not understood what "USEPA Level 3 data validation" means. Data validation should be performed using Region 2 Data Validation SOPs available at: http://www.epa.gov/region02/qa/documents.htm.

Response: this language has been replaced with a cross-reference to the data validation procedures in the QAPP amendment. The QAPP amendment references the Region 2 Data Validation SOPs.

#### Comment 3: QAPP

a. Provide an organization chart that shows reporting relationships between all organizations involved in the project, including the lead organization and all contractors and subcontractors.

Response: an organization chart has been included in Section 2.1 of the QAPP amendment.

b. Section 7.1 - This section references Table A-4 for reporting and detection limits for Method TO-15, however, these limits are shown on Table 4, Section 11.1. Please correct this discrepancy.

Response: the tables have been renumbered for consistency; references to the reporting limits are cross-referenced to the same table, which is now Table A-3.

c. Section 8.2 – It is stated that the "...data will undergo a data summary validation..." This should be further explained. The data obtained during this study should be validated using EPA Region 2 Data Validation SOPs or other equivalent criteria. The chosen Data Validation criteria should be described.

Response: the QAPP Amendment has been revised to make reference to the USEPA Region 2 Data Validation SOPs.

Response to Comments, e-mail from USEPA Region 2, March 13, 2006 (Sampling Locations Technical Memorandum)

Comment 1: No indoor air sampling is proposed at Building 11, the bank, because of weekend security issues. Due to very site specific conditions associated with the construction of the offices at 115 River Road and the impacts of these on vapor intrusion, Honeywell should provide any site specific information regarding ventilation rates, air exchanges, HVAC system details and other building specific issues that may influence the potential for vapor intrusion at this building so that EPA can evaluate the possibility of additional indoor air sampling in the future during a time when the building is accessible.

Response: information will be developed on Building 11 characteristics and documented in the post-sampling technical memorandum (see Table 6 of the revised workplan).

Comment 2: Ambient sampling is proposed, but the locations range from 0.5 miles to 1.8 miles from 115 River Road. It is unlikely that any ambient sources that might influence indoor air concentrations at 115 River Road would be detected at such a large distance. EPA recommends relocating these samples to a closer proximity to the building in order to identify and characterize any ambient sources. For example, with the relatively new construction and asphalt at the former Celotex property, it is possible that continuing offgassing of low molecular weight PAHs may be a source to indoor air at 115 River Road. However, no proposed ambient locations would identify these possible sources. One potential location for ambient samples would be on the Quanta property; it would be between the development at the former Celotex property and the building at 115 River Road and would not be in an area accessible to trespassers. Also, placing one ambient sample on the Quanta property near the Hudson River may identify possible vapors that would be present during low tide, while placing another ambient sample on the Quanta property near River Road may serve to identify the contribution of vapors from traffic. EPA is not convinced that the proposed locations would adequately identify any ambient sources.

Response: ambient samples have been relocated consistent with these comments. Three locations are near the 115 River Road building, and are intended to evaluate potential air quality impacts associated with nearby new construction, potential emissions from Hudson River sediments under low-tide conditions, and traffic on River Road. The discussion is presented in Section 4.3 of the revised workplan, Table 6 and Figure 3,

Comment 3: Hand drawn figure of the Building 8 Basement: The proposed subslab locations in the basement appear to be adequate, with one exception. EPA believes that this is the basement in which the cement floor was wet. If so, please ensure that one of the subslab samples are located as close to this area of the wet floor as possible.

Response: one of the subslab samples is located near the subject area where moisture was observed on the floor.

Comment 4: Hand drawn figure of the Building 10 Basement: The presents of a sump in this basement suggests that this may be an appropriate location for an indoor air sample.

The information that would be gathered by collected both a subslab sample and an indoor air sample from this basement would provide a comprehensive picture of the fate and transport of vapors into the building and would potentially identify the sump as a mechanism of transport for the vapors. EPA recommends adding an indoor air sample to the basement of Building 10.

Response: an indoor air sample will be placed in the Building 10 basement, located adjacent to the sump (see Table 6 and Section 4.2 of the revised workplan for description of this sample).

# Vapor Intrusion Evaluation Work Plan for the Quanta Resources Site, Edgewater, New Jersey

Prepared for USEPA Region II

On behalf of

Honeywell

101 Columbia Rd. Morristown, N. J.

Revised March 2006

Prepared by CH2MHILL Parsippany, NJ

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# Acronyms/Abbreviations

μg/kg microgram per kilogram

 $\mu g/L$  microgram per liter

ASTDR Agency for Toxic Substances and Disease Registry

bgs below ground surface

BTEX benzene, toluene, ethylbenzene, and xylenes

CFR Code of Federal Regulations

CLP Contract Laboratory Program

COI constituent of interest

DNAPL dense nonaqueous phase liquid

DOT Department of Transportation

FS feasibility study

GC/MS Gas Chromatography/Mass Spectrometry

HDNL Health Department Notification Level

HVAC heating, ventilation, and air conditioning

IATA International Air Transport Association

IASL Indoor Air Screening Levels

LCS laboratory control sample

LNAPL light nonaqueous phase liquid

mg/kg milligram per kilogram

mL milliliter

MRL minimal risk level

NJDEP New Jersey Department of Environmental Protection

NJDHSS New Jersey Department of Health and Senior Services

PARCC precision, accuracy, representativeness, completeness, and comparability

PCE tetrachloroethylene or tetrachloroethene

PRG preliminary remediation goal

QA quality assurance

QAPP quality assurance project plan

QC quality control

Quanta Quanta Resources

RAL Rapid Action Level

RI remedial investigation

RPD relative percent difference

RSI removal site investigation

SOP standard operating procedure

TCE trichloroethylene

USEPA United States Environmental Protection Agency

VOC volatile organic compound

# Introduction

This work plan has been prepared to evaluate potential vapor intrusion pathways at the Quanta Resources Site (the "site")¹, located in Edgewater, Bergen County, New Jersey. Investigation procedures will be conducted in accordance with this work plan to identify and evaluate potential vapor intrusion exposure pathways, if any, at the 115 River Road Property (former Spencer Kellogg buildings), located to the south of the Quanta Resources property. This document presents the data collection activities proposed to support the vapor intrusion pathway evaluation as outlined in the General Scope of Work submitted on January 12, 2006 and the U.S. Environmental Protection Agency (USEPA) comment letter dated December 5, 2005. The results from this evaluation will be used to identify potentially complete vapor intrusion pathways into the Former Spencer Kellogg building, if any, and to identify appropriate actions to address potentially complete vapor intrusion pathways, if needed.

Currently, a remedial investigation/feasibility study (RI/FS) is being conducted at the site in accordance with an Administrative Order on Consent with USEPA Region 2. This Vapor Intrusion Evaluation Work Plan is being submitted to the USEPA and the New Jersey Department of Environmental Protection (NJDEP) pursuant to the RI/FS, as requested by the two agencies.

## 1.1 Purpose and Scope of this Evaluation

This work plan focuses on potential vapor intrusion pathways along the southern boundary of the site, at the (115 River Road property (former Spencer Kellogg building)). Potential vapor intrusion pathways may exist on the remaining portion of the site. However, the Quanta Resources property is unoccupied, and contains no buildings. As a part of the RI/FS, an evaluation will be conducted of potential vapor intrusion pathways assuming possible future use or uses of the site. A separate work plan will be prepared for the vapor intrusion evaluation of the remaining portion of the site.

## 1.2 Regulatory Guidance for Vapor Intrusion Evaluation

This vapor intrusion evaluation will be conducted using the following regulatory guidance documents:

 Agency for Toxic Substances and Disease Registry (ATSDR). 2006. Minimal Risk Levels (MRLs) for Hazardous Substances (accessed January 8, 2006) http://www.atsdr.cdc.gov/mrls.html

<sup>&</sup>lt;sup>1</sup> As defined in the Administrative Order on Consent (AOC) II-CERCLA-2003-2012, the Quanta Resources Superfund Site includes the former Quanta Resources property, located on River Road in Edgewater, New Jersey, and any areas where contamination from the property has come to be located. The current extent of the Quanta Resources property (referred to herein as the "Quanta Resources property") refers to Block 95, Lot 1 as defined on the Borough of Edgewater, New Jersey tax map.

- New Jersey Department of Environmental Protection (NJDEP). 2005. New Jersey Vapor Intrusion Guidance, October 2005 (including updated screening tables, March 2006). http://www.nj.gov/dep/srp/guidance/vaporintrusion/vig.htm
- U.S. Environmental Protection Agency (USEPA). 1999. Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS).
- U.S. Environmental Protection Agency (USEPA). 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). November 29.
- U.S. Environmental Protection Agency (USEPA). 2004. Draft Standard Operating Procedure (SOP) for Installation of Sub-Slab Vapor Probes and Sampling Using EPA Method TO-15 to Support Vapor Intrusion Investigations.
- U.S. Environmental Protection Agency (USEPA). 2004. Region 9 Preliminary Remediation Goals http://www.epa.gov/region9/waste/sfund/prg/index.html
- U.S. Environmental Protection Agency (USEPA). 2006. Integrated Risk Information System (IRIS) http://www.epa.gov/iris (accessed January 8, 2006).

In addition, this work plan and attached quality assurance project plan () Amendment has been developed using guidance provided by USEPA Region 2 and the NJDEP.

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# **Preliminary Conceptual Site Model**

The following preliminary conceptual site model compiles information for characterizing potential vapor intrusion pathways in the vicinity of the 115 River Road building. This preliminary conceptual site model depicts sources of chemicals, migration pathways, potential receptor populations and potential exposure pathways from the chemical sources to the receptors. This conceptual site model has been developed to identify potential data needs related to vapor intrusion pathways that will be addressed through sampling and analysis. As additional data are collected, they will be incorporated into this preliminary conceptual site model to refine understanding of potential current and future vapor intrusion exposure pathways.

## 2.1 Site Background and Setting

The site is located within an industrial/commercial area in Edgewater, New Jersey (zip code 07020, Bergen County) (Figure 1). The property is bordered by the Hudson River to the east, the Edgewater Enterprises (former Celotex) and Lustrelon properties to the north, 3Y, LLC property to the west, and 115 River Road (former Kellogg Spencer) to the south. The former Unilever property is located south of the 115 River Road property. The Quanta Resources property is currently vacant and contains numerous exposed concrete tank and building foundations, a former oil/water separator, a wood bulkhead, and wooden docks along the Hudson River. The operations at the Quanta Resources property were as follows:

- 1876 1896: Fletcher Chemical Works
- Coal tar distillation plant operations
- Manufacturer of coal tars, asphalt, roofing materials
- 1896 1930: Barrett Manufacturing
  - Coal tar distillation plant operations
- 1930 1974: Allied Signal Inc.
- Coal tar distillation plant operations
- 1974: Frola & Van Dohln lease to ERP
- Storage and recycling of oil
- 1980-1981: Leased to Quanta Resources Corporation
- Storage and recycling of oil

The NJDEP ceased facility operations at the Site in 1981 after it was discovered that large quantities of oil were present in storage tanks at the facility, including some with concentrations of PCBs. At that time the site contained 61 aboveground storage tanks (ASTs) with a capacity of 9 million gallons, along with at least 10 underground storage tanks (USTs). On October 6, 1981, Quanta Resources Corporation filed for bankruptcy, after which the property was no longer in use. Periodic flooding of the Hudson River, equipment failures, freezing and thawing of pipes and tanks, rusted values valves and seams, and the lack of containment structures, and the migration of NAPL resulted in releases. NJDEP requested that USEPA address site

contamination pursuant to Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). From 1984 to 1988, USEPA removed the ASTs, USTs, septic tanks, underground piping, 1.35 million gallons of oil, and over 1.5 million gallons of coal tar. Activities at the Quanta Resources property may have impacted adjacent properties, including the 115 River Road property.

#### 115 River Road Property

Three distinct buildings are present on the 115 River Road property (Figure 2). The main 115 River Road building consists of two attached buildings (older building and newer Pier building) that are approximately 800 feet long and between 30 and 60 feet wide. The original western portion of this building located between River Road and the Hudson River is approximately 500 feet long and reportedly dates back to the turn of the 20th Century (referred to in Figure 2 as the 'Main 115 River Road building'). This building is constructed of brick and contains two distinct basements.

The second office building consists of an approximately 300 foot long expansion of the main building which extends over the Hudson River. It appears to have been constructed in the 1990s (based on the dates of available design drawings) and is referred to in Figure 2 as the 'pier building'. The main building is 2- and 3-stories, and approximately 30 to 40 feet in height. The newer pier building has aluminum frame construction with insulated glass and corrugated metal siding and is elevated approximately 10-15 feet above the Hudson River. The inhabited office spaces are elevated approximately 12 feet over an open-air parking lot.

A third, smaller two story, brick building (Building 12), approximately 100 feet by 25 feet, is located to the north of the main 115 River Road building. The remainder of the approximate 3-acre upland area consists of parking area paved with either asphalt or concrete. Building 12 is a 2-story, brick building approximately 30 feet in height. In that building, the inhabited office spaces are elevated over a semi-enclosed parking lot.

The main building is divided into 10 different tenant-occupied "buildings", numbered from east to west as Buildings 2 through 11 (Figure 2). Basements are located under Building 10 and Building 8; the Building 8 basement also appears to extend partly under Building 7.

Historically the 115 River Road property was used in the manufacture of linseed oil and for other industrial operations.

# 2.2 Overview of Vapor Intrusion Pathway

Volatile organic compounds (VOCs) located in subsurface soils or in groundwater can volatilize, migrate through soil gas and subsequently can be transported into indoor spaces, potentially producing inhalation exposure. VOCs in soil gas will migrate and spread out by vapor diffusion. The VOC flux through soil gas is proportional to the concentration gradient (i.e., the difference in concentration between high-VOC and low-VOC concentration areas).

The tendency for a VOC to migrate by vapor diffusion is reflected by its air diffusion coefficient. Air-filled porosity in soil influences VOC vapor migration; air-filled porosity is in turn affected by soil properties such as total porosity, clay or silt content, and moisture content. Vapor diffusion of a VOC in soil decreases with low air-filled porosity and high moisture content and

low air diffusion coefficient. VOCs dissolved in groundwater will partition into the overlying soil gas. The tendency for a VOC to migrate from groundwater to soil gas is reflected by its Henry's Law coefficient. The larger the Henry's Law coefficient, the more likely it is that a chemical will migrate into soil gas from groundwater. The ability for a VOC to enter soil gas from groundwater is also influence by the capillary zone immediately above the water table. VOC diffusion through the capillary zone will be small when the capillary zone is highly water-saturated.

Molecular diffusion moves the VOC towards the soil surface until it reaches the zone of influence of a building. Convective air movement within the soil column transports the vapors through cracks between the foundation and the basement slab floor. This convective sweep effect is induced by a negative pressure within the structure caused by a combination of wind effects (wind pressure on the building exterior) and the stack effect due to building heating and mechanical ventilation. The concentration in indoor air resulting from VOC intrusion into a structure resulting from pressure-driven air flow is related to the building volume (length, width, and height) and the building ventilation rate (numbers of air changes per hour).

#### 2.3 Potential Sources

Potential sources of constituents for vapor intrusion along the southern border of the Quanta Resources property in the vicinity of the 115 River Road buildings are oil-like product in soil and shallow groundwater, dissolved-phase VOCs in groundwater, total VOCs in soil, materials currently stored in the buildings, and potentially, chemicals released from historic industrial operations at the 115 River Road property. The surface fill at the site consists of 9 to 18 feet of fine to medium grain sand containing some silt, cinders, brick, wood, gypsum, and concrete debris. Groundwater at the Quanta site occurs within the unconsolidated fill at a depth of 3 to 7 feet below ground surface (bgs). The soil and groundwater conditions in this area were based on cross-sections to the north and south of the 115 River Road property (GeoSyntec, 2000). A cross-section on the east side of the 115 River Road property shows fill containing product at a depth of 8 to 10 feet below grade (beneath the water table), at the southern boundary of the Quanta Resources property. A recent survey of product in monitoring wells, conducted in December 2005, observed 4 feet of product in monitoring well MW-107, located to the south of the 115 River Road property.

Descriptions of the VOCs detected in soil and groundwater were drawn from the Removal Site Investigation (RSI) (GeoSyntec, 2000). These descriptions are presented in the following paragraphs.

Characterization of the free product was conducted during the RSI using Rapid Optical Screening Tool (ROST<sup>TM</sup>). The borings on the southern boundary of the Quanta Resources property, closest to the 115 River Road property, reportedly contained heavy-end and undifferentiated hydrocarbon product. A survey of nonaqueous free product in monitoring wells was conducted in October and December 2005. None of the groundwater monitoring wells in the vicinity of the 115 River Road property contained light nonaqueous phase liquid (LNAPL), though a sheen was observed on the surface of the water column in each well. Dense nonaqueous phase liquid (DNAPL) was observed in some wells along the southern border of the Quanta Resources property, characterized as thick, black product.

VOCs detected in soil at the Quanta Resources property principally consisted of benzene, toluene, ethylbenzene, and xylenes (BTEX), present at total concentrations around 1 milligram per kilogram (mg/kg) or less along the southern boundary of the site (see Table 1). Acetone was also detected in one soil boring south of the 115 River Road property at concentrations below 1 mg/kg. Soil data for polyaromatic hydrocarbon (PAH) constituents acenaphthene, benzo(b)fluoranthene, chrysene, fluorene, and naphthalene are being considered because USEPA's draft vapor intrusion guidance (USEPA, 2002) identifies them as sufficiently volatile to be a constituents of interest in evaluating vapor intrusion. These constituents were generally detected in soil at concentrations below 10 mg/kg (most below 1 mg/kg), except at location MW-105 (~250 feet east of the building) at a depth of 5 feet in soil where concentrations for all five constituents ranged from 1,100 to 3,400 mg/kg (Table 1). These PAH constituents were also detected in soil below the water table (18 feet bgs) at concentrations ranging from 49 to 1,900 mg/kg at boring MW-106, which is located approximately 70 feet southeast of the building.

Soil gas data have not been collected at the site.

VOCs detected in groundwater along the southern boundary of the Quanta Resources property site principally consist of BTEX (see Table 2) with one detection of chlorobenzene. Total BTEX concentrations north of the 115 River Road property (on Quanta Resources property) range from 14,000 to 23,000 micrograms per lited ( $\mu$ g/L). South of the 115 River Road property, concentrations are considerably lower, ranging from 7.7 to 26.3  $\mu$ g/L, except at monitoring well MW-107 where at 9,100  $\mu$ g/L the concentration is still considerably lower than to the north (Table 2). Similarly, naphthalene concentrations in groundwater are higher north of the 115 River Road property (16,000 to 23,000  $\mu$ g/L) than south of the property (12 to 11,000  $\mu$ g/L).

Other constituents have been detected in groundwater at the Quanta Resources property, but not in proximity to the 115 River Road property. These consist principally of chlorinated VOCs including tetrachloroethylene (PCE), trichloroethylene (TCE), 1,1,1-trichloroethane, 1,1-dichloroethane, cis-1,2-dichloroethylene, and chloroform.

## 2.4 Potential Migration Pathways

The presence or absence of potential migration pathways from constituent sources in soil and groundwater to indoor air involves consideration of the following factors:

- A shallow water table (approximately 7 feet bgs) underlies the 115 River Road property, reducing the distance that VOCs in groundwater must diffuse to reach the building foundations. While concentrations detected in groundwater are higher than USEPA screening levels, it is not certain how representative the groundwater results are with respect to vapor intrusion potential. Groundwater concentrations at the top of the water-bearing zone (within a few feet of the water table) have the highest potential for volatilization into overlying soil gas (Rivett, 1995; USEPA, 2002). The monitoring wells onsite are screened throughout the water-bearing zone, so that the results are not representative of groundwater concentrations at the top of the water-bearing zone. As described below, sampling is proposed to evaluate the potential for volatilization of VOCs from groundwater to the overlying soil gas.
- LNAPL (with the exception of some sheen) has not been observed in groundwater. DNAPL
  has been observed in groundwater, several feet beneath the water table, in different portions

of the Quanta Resources property. Soluble constituents in DNAPL could potentially dissolve, producing concentrations of constituents (including VOCs) in groundwater. As discussed previously, it is not certain how representative the groundwater results are with respect to vapor intrusion potential.

- Discontinuous pockets of nonaqueous phase product have been observed in soil in different portions of the Quanta Resources and former Unilever Research properties. In addition, soil sampling has identified VOCs in soil, based on total soil concentration (micrograms per kilogram [µg/kg] in soil). The locations sampled were not close to the 115 River Road property, so it is not known if VOCs and/or free product in soil contribute to potential vapor intrusion. In addition, soil gas sampling has not been conducted at the site. As described below, sampling is proposed to further evaluate the potential for vapor intrusion pathways to be present from VOCs in shallow soil in proximity to the building.
- Building conditions that may influence vapor intrusion potential are currently not known. As described below, the sampling approach will first involve conducting a building walkthrough survey to better characterize the building and refine selection of proposed sampling locations. During this walkthrough survey, the building will be observed for the presence of conditions generally thought to increase the potential for vapor intrusion, including floor cracks, spaces between floor-wall joints, utility (gas, electric, sewer or water) intrusions in floors and walls, floor drains or sumps, and basements. Also, it is not known if subsurface utilities underlying the building potentially represent preferential pathways for the migration of VOCs. During the walkthrough survey, information will be obtained, if available, to better under the location and depths of subsurface utilities.

### 2.5 Potential Receptors

Although specific construction details of the three buildings at the 115 River Road property have not be obtainable, a building layout provided by the property owners indicates that the 115 River Road building is a large brick building that has been renovated and is currently being used for office suites, a child care center, and a parking garage (NJDOHSS, 2002). Observation of an aerial photograph of the building suggests that approximately the eastern third of the building is on a newly reconstructed pier over the Hudson River. The "ground level" of this pier reportedly consists of an open-air parking garage with the upper levels of the structure occupied by office space. Vapor intrusion pathways are less likely to be present in this portion of the building, because of a lack of contact with the subsurface. The smaller two-story "brick" building contains an open-air parking area on the ground level with offices located above.

Populations within the building are expected to consist of building management and maintenance staff, and tenants leasing space within in the building. Children are also present at the day care center.

# Constituents of Interest and Response Levels

### 3.1 Identification of Constituents of Interest

The constituents of interest (COIs) represent the analytes detected at the site that may warrant further evaluation for potential vapor intrusion. Not all of the analytes detected at the site warrant evaluation for vapor intrusion potential. Many are not sufficiently volatile to migrate appreciably from affected media by diffusion.<sup>2</sup> Others have not been detected in soil or groundwater samples located in close proximity (i.e,. within 300 feet) to the 115 River Road property.

The initial sampling event conducted as part of this vapor intrusion evaluation (described below) will evaluate a range of target analytes to identify the constituents of interest. The target analyte list is discussed in further detail in the QAPP Amendment, presented in Appendix A. A arget nalyte ist of 60 constituents will be analyzed as part of the initial sampling event. However, the chemicals most likely to represent constituents of interest for vapor intrusion are BTEX and naphthalene. As discussed previously in the conceptual site model, these analytes have been detected in samples collected near the 115 River Road property and were found at concentrations approaching or exceeding USEPA target concentrations in groundwater (Table 2).

### 3.2 Response Levels

Data collected during this vapor intrusion evaluation will be used to identify further activities or responses, if any are warranted. The need for specific activities or responses will be determined on a case-by-case basis, depending upon the sampling results. However, it is anticipated that there are a range of potential responses that can be considered, including the following:

- No further investigation required.
- Perform periodic follow-up air monitoring.
- Perform further investigation in specific areas to confirm presence of concentrations in indoor air, to identify potential constituent sources or identify potential migration pathways.
- Discontinue sampling at specific sampling locations, discontinue sampling for specific constituents of interest, and/or terminate implementation of selected mitigation measures.
- Evaluate the need to implement mitigation measures within specific portions of the building.

<sup>&</sup>lt;sup>2</sup> USEPA's draft vapor intrusion guidance (USEPA, 2002) provides a list of constituents considered sufficiently volatile to warrant evaluation for vapor intrusion. The constituents of interest discussed in this work plan have been identified based on the list presented in USEPA's draft guidance document.

• Identify conditions where temporary relocation of occupants from selected locations within the building may be advisable during implementation of interim mitigation measures.

The need for further responses will be based on comparison of indoor air and subslab sampling results with response levels. The process for evaluating indoor air and subslab sample results is based on decision matrices developed by USEPA Region 2 (USEPA, 2004). These decision matrices are presented in Tables 3a (for carcinogenic constituents) and 3b (for noncarcinogenic constituents). The response levels in indoor air and subslab samples for the target analyte list are shown in Table 4.

The response levels in indoor air, shown in Table 4, were derived from USEPA Region 9 Preliminary Remediation Goals (PRGs) in air, or New Jersey Department of Environmental Protection (NJDEP) Indoor Air Screening Levels (IASLs) (NJDEP, 2005; NJDEP, 2006), whichever value was more conservative. Use of USEPA Region 9 PRGs is based on guidance for vapor intrusion workplan preparation provided by USEPA Region 2. Subslab response levels were derived from the indoor air response levels using an attenuation factor of 0.1, as recommended by USEPA (USEPA, 2002).

The specific sequence of activities, and timing of those activities, in response to detected levels in indoor air or subslab samples corresponding to the decision matrices (Tables 3a and 3b) are discussed in the following paragraphs.

NO FURTHER ACTION – Following consultation with the regulatory agencies, indoor air and subslab for the constituents of interest (COIs) may be discontinued at the sampling locations where indoor air and subslab concentrations from the first sampling event, and indoor air concentrations from the second sampling event, fall within the specified response levels in Tables 3a and 3b, or indoor air concentrations are lower than background levels in ambient air (measured as described in Section 4.4).

MONITOR – Following consultation with the regulatory agencies, perform indoor air sampling semiannually (one warm season; one cold season sample) at locations where concentrations of COIs in indoor air and subslab samples fall within the specified response levels in Tables 3a and 3b, or indoor air concentrations are higher than background levels in ambient air (measured as described in Section 4.4). Reevaluate indoor air concentration data after two additional sampling events to determine if frequency of sampling needs to be revised or sampling discontinued.

INVESTIGATE – Following consultation with the regulatory agencies, conduct further investigation (possible investigation approaches may include subslab sampling, "crack-and-crevice" investigation, evaluation of building heating, ventilation and air conditioning (HVAC) system, conduct building survey for potential indoor sources) to better identify potential sources and pathways; reevaluate indoor air concentration data to determine if frequency of and locations for indoor air sampling need to be revised. Identify appropriate engineering controls (for example, seal cracks and crevices, install subslab depressurization system, rebalance HVAC to overpressurize building) to reduce indoor air concentrations, for prompt implementation if needed. Focus these investigation activities locations where concentrations of COIs in indoor air and subslab samples fall within the specified response levels in Tables 3a and 3b.

PURSUE REMEDIATION – Following consultation with the regulatory agencies, implement engineering controls (for example, seal cracks and crevices, install subslab depressurization system, rebalance HVAC to overpressurize building) to reduce indoor air concentrations; evaluate and implement interim measures for subsurface sources.

In addition, indoor air sampling results will be compared with NJDEP Rapid Action Levels (RAL) and Health Department Notification Levels (HDNL) (NJDEP, 2005). The available RALs and HDNL values are presented in Table 5. Methods for calculating RALs and HDNLs for other target analytes if needed are also shown in Table 5.

RALs represent trigger levels for the initiation of prompt action at occupied buildings to further investigate the vapor intruion pathway and/or minimize impacts to building occupants through the implementation of an interim remedial measure (IRM) (NJDEP, 2005). As described previously, these measures may include engineering controls (for example, seal cracks and crevices, install subslab depressurization system, rebalance HVAC to overpressurize building) to reduce indoor air concentrations.

HDNLs, when exceeded in occupied buildings, indicate the need for the NJDEP to inform the local and/or state health departments about the site and the associated vapor intrusion related indoor air concentrations for further evaluation and possible emergency actions (NJDEP, 2005). If concentrations of COIs in indoor air at a location are at HDNL levels in any indoor air sampling event, indoor air will be promptly resampled, and analyzed on a rush basis. In addition, the building owners/management will be notified to discuss temporary relocation of occupants.

# Sampling Rationale

USEPA's draft vapor intrusion guidance (USEPA 2002) identifies a tiered process for evaluating potential vapor intrusion pathways. These tiers are:

- Tier 1-Primary Screening: quickly identify if a potential pathway exists at a site.
- Tier 2-Secondary Screening: compare measured or reasonably estimated concentrations of constituents in various media (i.e., groundwater) to conservative risk-based screening values.
- Tier 3–Site-Specific Pathway Assessment: this is a more detailed evaluation of the vapor intrusion pathway, typically involving direct measurement of vapor intrusion potential.

The sampling approach presented in this work plan reflects a Tier 3 site-specific pathway assessment. It focuses on areas both inside and outside of the office building. This approach consists of gathering several discrete types of data intended to provide an overarching picture of conditions at the site.

As discussed in the previous section, the data collection activities are connected with specific responses; these responses are intended to gather further data, and/or initiate mitigation measures to reduce indoor air concentrations, if needed (Table 3).

The sampling tasks consist of a building survey, soil gas sampling, sub-slab gas sampling, indoor air sampling, and outdoor ambient (background) air sampling. A preliminary description of the sampling approach is presented in Table 4. Note that this approach may be modified based on the purpose for each of these tasks, and uses for the data generated from them are discussed in the following paragraphs.

### 4.1 Building Surveys

The walkthrough survey of the property buildings was conducted on February 27 and 28, 2006. The objectives of the survey included: (1) identification of potential storage and indoor uses of chemicals or products; (2) evaluation of foundation and basement conditions to identify potential intrusion points and potential utility clearance issues; and (3) conduct of a preliminary evaluation of HVAC operation, and identification of outdoor air intakes.

The results from the building surveys will be used as follows:

- Identify and document potential indoor sources that might affect indoor air sampling results.
- Identify potential locations for sub-slab samples; also identify potential utility clearance
  problems with those locations. Proposed locations for subslab samples are described below
  in Section 4.2.

- Identify potential locations for indoor air samples. Proposed locations for indoor air samples are described below in Section 4.2.
- Evaluate overall building airflow characteristics; identify areas that might be either
  positively or negatively pressurized; observe if feasible the amount of outside air being
  delivered to indoor spaces.

# 4.2 Synoptic Subslab and Indoor Air Sampling

Subslab and indoor air samples will be collected in accordance with QAPP Amendment (Appendix A) from the 115 River Road buildings (the main building, the pier extention and Building 12). The proposed number and locations for the subslab indoor air samples have been identified from the walkthrough survey. The subslab and indoor air samples will be collected synoptically. This differs from USEPA's draft vapor intrusion guidance, which recommends collecting subslab samples prior to indoor air samples. However, the indoor air and subslab samples will be collected together to facilitate calculation of attenuation factors. Also, synoptic<sup>3</sup> sampling will minimize disruption of building activities and will accelerate data collection and interpretation.

The subslab and first indoor air sampling event will be performed during the "cold" season (i.e., before the end of March). A second indoor air sampling event will be conducted during the warm season (i.e., between April and October).

The subslab probes will remain in place for follow-up sampling if needed. Indoor air, subslab and background air sampling data will be reviewed prior to making a decision to abandon the subslab probes.

The results from the indoor air and subslab sampling events will be evaluated as described in Section 3. In addition, subslab sampling results will be used to better identify constituents of interest, which may require further evaluation for vapor intrusion potential. The proposed indoor air and subslab sampling locations are described in the following sections.

### **Proposed Sampling Locations**

As discussed with USEPA during the property visit on February 27, 2006, the sampling locations for the upcoming sampling event focuses on the basements, which represent potential conduits for vapor intrusion, and the indoor spaces overlying the basements. Subslab samples will be collected from beneath the basement foundation and indoor air samples will be collected from inhabited spaces, as noted in attached Table 6. In addition, subslab samples will be collected from the Building 12 parking lot and indoor air samples will be collected from Building 12 inhabited spaces. Finally, indoor air samples will be collected from inhabited spaces of the pier building.

Nineteen indoor, ten subslab and seven ambient samples (including duplicates) are proposed. The descriptions of the locations are presented in Table 6. Hand-drawn sketches of the Building 8 and Building 10 basements showing the proposed locations of the subslab samples are presented in Appendix B.

<sup>&</sup>lt;sup>3</sup> This signifies that indoor air and subslab samples will be collected at the same time, in order to provide a snapshot of both indoor and subsurface conditions at the building.

# 4.3 Ambient (Outdoor) Air Sampling

Ambient air samples will be collected from outdoor locations. The objectives for collecting ambient air samples are: 1) evaluate ambient air quality close to the 115 River Road building, to identify and characterize ambient sources potentially affecting air quality in the building; 2) evaluate local ambient air quality to supplement literature values for typical ambient levels of constituents in urban air.

The results from the ambient air sampling and literature values will be used to develop site-specific background contaminant values in air. The site-specific background values in air will be used along with risk-based concentrations to evaluate indoor air sampling results and assist in refining the list of COIs. The purpose for the ambient samples from the 115 River Road building roof will be to identify the potential for entry of VOCs from ambient air into the building.

The proposed outdoor air sampling locations are identified in Table 6. Three locations are near the 115 River Road building, and are intended to evaluate potential air quality impacts associated with nearby new construction, potential emissions from Hudson River sediments under low-tide conditions, and traffic on River Road. Two proposed locations are outside the footprint of impacted groundwater from the Quanta Resources Site. In addition, two ambient air samples will be collected from the roof of the 115 River Road building, near outdoor air intake vents. All of these outdoor air samples will be collected during the synoptic indoor air and subslab sampling event. A figure showing the proposed locations of the outdoor air sampling locations is presented in Figure 3.

# Sampling and Analysis Procedures

This section describes the sampling and analysis procedures that will be used to implement the sampling approach described in the previous section.

## 5.1 Building Surveys

The walkthrough survey of the property buildings, conducted February 27-28, 2006 consisted of a physical site visit to observe physical building construction features and the HVAC system, identify locations of subsurface utilities, and review use and storage of chemicals within the building. A building survey questionnaire is presented in the QAPP Amendment (Appendix A)

The results from this survey have been used to recommend the locations of the indoor air and subslab samples as part of the synoptic indoor air and subslab sampling event (see Section 4.2).

## 5.2 Subslab Vapor Sampling

Subslab vapor sampling will be performed at locations identified during the building survey. A SOP is presented in the QAPP Amendment (Appendix A) for installation of subslab sample probes and vapor sampling using SUMMA<sup>TM</sup> canisters. Subslab sample probes are installed flush with the building floor using an industrial hand drill with concrete masonry drill bits. The probe hole is sealed at the floor surface with water-based mortar and leak checked using helium to ensure that ambient air is not introduced along with the subslab vapor sample. Samples are collected using SUMMA<sup>TM</sup> canisters with flow controllers, as described in the QAPP Amendment.

# 5.3 Air Sampling (Indoor and Ambient)

Air samples will be collected over a 24-hour period using SUMMA™ canisters equipped with flow controllers. SOPs for SUMMA canister sampling and flow controller calibration are presented in the SOP. Indoor air samples from occupied spaces will be collected from a height between 2 and 5 feet above the ground (roughly, breathing zone height). The indoor air sample collected from the Building 10 basement location will be placed on the floor adjacent to a sump. The field sampling team will, whenever possible, note activities in the sampling area (such as chemical use or storage) that may potentially influence the indoor air sampling results. Outdoor and indoor air samples will be collected concurrently. Barometric pressure and temperature during the sampling event will be measured on site during sampling using portable instruments.

# 5.4 Laboratory Analysis

All air samples (soil gas, subslab, indoor air, ambient air) collected in SUMMA™ canisters will be analyzed following USEPA Method TO-15, Determination of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters and Analyzed by Gas Chromatography/ Mass Spectrometry.

Analyses will be performed by Columbia Analytical Services, Simi Valley, California. This laboratory is certified with the NJDEP through the National Environmental Laboratory Accreditation Program. The laboratory will provide data of comparable quality and documentation that are consistent with the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), June 2005

(<u>http://www.epa.gov/fedfac/documents/qualityassurance.htm</u>). The measurement quality objectives for analyses using Method TO-15 GC/MS and GC/MS-SIM analysis are described in the OAPP Amendment (Appendix A).

The laboratory will supply certified clean SUMMA canisters with individual tracking numbers and certified clean and calibrated flow regulators. Turnaround time that will be requested for the analysis will be 14 days.

The target analyte list and associated reporting limits are presented in Table A-3 in the QAPP Amendment. The reporting limits are intended to achieve the lowest risk-based levels described in Table A-1 in the QAPP Amendment.

#### **SECTION 6**

# Occupant Notification and Obtaining Access

The building owners/managers of the 115 River Road building have been contacted to arrange access to the building for performing sampling activities. The sampling activities will be conducted during a weekend to minimize disruption to building activities.

Prior to initiating any sampling activities, the proposed sampling locations and sampling methods were discussed with the building owners/managers. Particular attention was be given to describing the subslab sampling methods. The locations for these samples, particularly with regard to potential subsurface utilities, were be discussed with the building owners/managers prior to installation of the subslab probes. The building owners/managers have committed to clear each subslab sampling location before installation of the subslab probes.

#### **SECTION 7**

# Data Evaluation, Interpretation and Reporting

Results from the synoptic indoor air and subslab sampling, along with the results from soil gas and ambient (background) sampling will be evaluated as follows:

- Subslab and ambient air sampling results will be used to identify constituents of interest in indoor air.
- Indoor air results for constituents of interest will be compared with the response levels to
  identify further activities to investigate and, if needed, mitigate potential vapor intrusion
  pathways.
- Subslab and indoor air results for constituents of interest will be used to calculate attenuation factors. The attenuation factors will be used to refine the conceptual site model of the potential vapor intrusion pathways.

The results from the synoptic sampling event will be summarized in tables and posted on figures. These summary tables and figures will be provided to the building owners/ managers, USEPA and NJDEP. A cover letter for distribution to the building owners/managers will be prepared to provide a brief interpretive narrative of the results. This cover letter will accompany the data package (tables and figures) from the synoptic sampling event.

Summary tables will be provided to the building owners/managers, USEPA, and NJDEP after each of the follow up air sampling events.

Fourteen-day turnaround times will be requested from the analytical laboratory. Laboratory data will undergo validation as described in the QAPP Amendment before evaluation and reporting. It is anticipated that results will be available approximately 30 days following submittal to the analytical laboratory.

A technical memorandum summarizing the results from the synoptic sampling event will be prepared for submittal to USEPA, NJDEP and the building owners/managers. The results from this evaluation also will be incorporated into the RI/FS report for the site.

## **Schedule**

The following project schedule milestones are:

- February 27-28, 2006– Conducted building walkthrough survey.
- March 1, 2006–Start mobilization for field activities. Meet with building owners/ occupants
  to discuss building access. Start identifying potentially suitable locations for ambient
  (outdoor) air samples.
- March 9, 2006–Provide recommended subslab and indoor air sample locations based on walkthrough survey. (Technical Memorandum to USEPA and NJDEP).
- March 18-19, 2006-Conduct initial ambient (outdoor), synoptic indoor air and subslab, sampling events.
- April 20, 2006 (approximately)-results from synoptic sampling event will be available for distribution to building owners/managers, USEPA and NJDEP.
- May 15, 2006 (approximately)–Data from synoptic sampling event available for distribution to building owners/managers, USEPA and NJDEP.
- Mid-June 2006–Start mobilization for second indoor air and ambient air sampling event. Conduct sampling event.
- August 2006-Data from second air sampling event available for distribution to building owners/managers, USEPA and NJDEP. Evaluate the need for further indoor and ambient sampling events.
- December 2006–Initiate semiannual indoor air (and ambient air) sampling, if needed.

## References

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U.S. Environmental Protection Agency (USEPA). 2002. *Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance)*. November 29.

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TABLE 1
Detected Concentrations of Constituents of Interest in Soil
Quanta Resources Site
Edgewater, NJ

			,	
Location Sample Depth	MW-105 (~250 feet E. of building)	` <b>-</b> /	CPT-24 (~150 feet SW. of building)	, ,
Analyte (mg/kg)	5 feet deep	19 feet deep	2.5 feet deep	4.5 feet deep
VOCs 5				Market Control of the
Acetone				
Benzene				
Toluene	3.6 J			1.2
Ethybenzene			0.14J	0.3 J
Xylenes			0.27J	1.6
Total VOCs	3.6	0	0.41	3.1
PAHS *	THE PROPERTY OF THE PARTY.			
Acenapthene	1200	0.56J	1.3	4.3
Benzo(k)fluoranthene	1100	0.19	1.2	1.7
Chrysene	2200	0.45J	2.8	6.3
Fluorene	1400	0.6	1.3	0.94J
Naphthalene	3400	1.4	5.2	1.2J

Location Sample Depth Analyte (mg/kg)	MW-106 (~70 feet SE. of building) 9 feet deep	MW-106 (~70 feet SE. of building) 14 feet deep	MW-106 (~70 feet SE. of building) 18 feet deep ∠	MW-106 (~70 feet SE. of building) 23 feet deep		
VOCs 7	TO THE PROPERTY OF		<b>第3</b> 年 1500年 1500年 1500年	<b>通常的企业。</b>		
Acetone						
Benzene	.15 J					
Toluene	.96 J		14.0 J			
Ethybenzene						
Xylenes						
Total VOCs	1.11	0	14	0		
PAHS	THE PROPERTY OF STREET	Control of the second				
Acenapthene	1.3J	0.63	350	11		
Benzo(k)fluoranthene	0.64	0.9	49	1.5		
Chrysene	2	2	110J	4.7		
Fluorene	1.2J	0.51J	310	9.2		
Naphthalene	3.6	0.79	1900	28		

<u>Location</u> <u>Sample Depth</u> Analyte (mg/kg)	MW-107 (~140 ft S. of building) 13 feet deep	MW-107 (~140 ft S. of building) 17 feet deep	MW-107 (~140 ft S. of building) 23 feet deep
VOCs*		和65年的公司 医克里克氏	
Acetone	0.77	0.66	
Benzene		0.47	1.5
Toluene		0.52 J	0.9
Ethybenzene			
Xylenes		-	
Total VOCs	0.77	1.65	2.4
PAHS:		CALL STANDS OF THE SHOP	M. 16 (4) (4) (4) (4) (4) (4) (4) (4) (4) (4)
Acenapthene	0.36J	0.36J	0.8
Benzo(k)fluoranthene			0.032J
Chrysene			0.14J
Fluorene	0.073J	0.31J	0.89
Naphthalene	3.7	1.6	1.9

Source: RSI Report, Geosyntec, 2000: Figs. 6-2 Total VOC in Soil.

TABLE 2
Detected Concentrations of Constituents of Interest in Shallow Groundwater
Quanta Resources Site
Edgewater, NJ

Location			
Sample Depth	MW-102	MW-103	
	(~ 90 ft N. of building)	(~100 ft N. of building)	Target Groundwater
Analyte (ug/L)			Concentrations (ug/L)
VOCs			
Acetone			2,080,000
Chlorobenzene			800
Benzene	14,000	2,200	11
Toluene	6,000	6,100	19,000
Ethybenzene	660	1,100	3,400
Xylenes	2,400	5,000	350
Total VOCs	23,920	15,600	
PAHS			
Acenapthene	870		NA NA
Chrysene	260		NA
Fluorene	940		NA_
Naphthalene	23,000	16,000	160
SVOCs			
Styrene	860	1,200	9,780

Location	100			T
Sample Depth	MW-106	MW-107	MW-108	Target Groundwater
į l	(~70 ft SE of building)	(~140 ft S. of building)	(~90 ft S. of building)	Concentrations
Analyte (ug/L)				(ug/L)
VOCs = 1				
Acetone			8.6	2,080,000
Chlorobenzene			1.5	800_
Benzene	6.3	5,300	3.1 <sub>-</sub>	11
Toluene	20	3,800	4.6	19,000
Ethybenzene				3,400
Xylenes				350
Total VOCs	26.3	9,100	17.8	
PAHS				
Acenapthene	100	230	10	NA NA
Chrysene	5.8			NA
Fluorene	72	92	. 2.3	NA
Naphthalene	1,200	11,000	12	160
SVOCs		<b>表决定任务工程的基本系统</b>		BRITIST TO SEE
Styrene				9,780

Notes:

Source: RSI Report, Geosyntec, 2000: Figs. 6-11 Total VOC in Groundwater Locations of the monitoring wells are relative to the Spencer Kellogg building.

NA - not applicable. Target soil gas concentration exceeds maximum possible vapor concentration at this soil gas to indoor air attenuation factor (pathway incomplete) (USEPA, 2002)

Target Groundwater Concentration Corresponding to Target Indoor Air Concentration Where the Soil Gas Concentration Where the Soil Gas to Indoor Air Attenuation Factor = 0.001 and Partitioning Across the Water Table Obeys Henry's Law (USEPA, 2002). Values have been updated using current USEPA toxicity values from IRIS when necessary.

Target concentration for benzene corresponds to a 1  $\times$  10<sup>-5</sup> target cancer risk. Target concentrations for other constituents corresponds to a noncancer hazard quotient (HQ) of one.

Table 3a Vapor Intrusion Matrix for Carcinogenic Chemicals Quanta Site, Edgewater, New Jersey

Subslab Soil Gas		Indoor Air Concentrations	
Concentrations	10 <sup>-6</sup> CR to <10 <sup>-5</sup> CR	10 <sup>-5</sup> CR to <10 <sup>-4</sup> CR	>10 <sup>-4</sup> CR
10 <sup>-6</sup> CR to <10 <sup>-5</sup> CR	NO FURTHER ACTION. Potentially site- related and within acceptable risk range.	2. INVESTIGATE - Potentially site-related and within acceptable risk range. Further investigation needed to identify a source or preferential pathway(s).	3. INVESTIGATE - May not be site- related. Further investigation needed to identify a source or preferential pathway(s).
10 <sup>-5</sup> CR to <10 <sup>-4</sup> CR	4. MONITOR - Probably site-related and within acceptable risk range. Potential for future exposures. Collection of additional data or further investigation may aid decision process, however reliablee decisions may be made with the inclusion of other factors, such as cost, which would suggest that pursuing remediation is warranted.	5. MONITOR or PURSUE REMEDIATION - Probably site-related with potential for exposures at concentrations approaching the upper-bound of the acceptable risk range. Collection of additional data or further investigation may aid decision process, however reliablee decisions may be made with the inclusion of other factors, such as cost, which would suggest that pursuing remediation is warranted.	6. INVESTIGATE or PURSUE REMEDIATION - Potentially site-related and above the upper-bound of the acceptable risk range. Further investigation needed to identify other potential sources or preferential pathway(s). If none found, remediation is the preferred option due to indoor air concentrations exceeding the upper-bound of the acceptable risk range.
	Probably site-related and within acceptable risk range, however further monitoring may be advisable prior to remediation to determine the future potential for vapor intrusion from elevated soil gas concentrations. Alternativelh, remediation could be performed to eliminate the potential for future expousres with potential cost savings by eliminating additional monitoring		9. PURSUE REMEDIATION - Probably site-related and above the upper-bound of the acceptable risk range. Elevated soil gas and indoor air could lead to future exposures with risks above the acceptable range.

Source: USEPA Region 2, 2004

CR - cancer risk

Notes:

Indoor air concentrations also will be compared with measured background concentrations (see Section 4.3) as well as 10<sup>-6</sup> or 10<sup>-5</sup> CR risk-10<sup>-6</sup> CR to <10<sup>-5</sup> CR - based indoor air response levels.

Indoor air concentrations also will be compared with measured background concentrations (see Section 4.3) as well as 10<sup>-5</sup> or 10<sup>-4</sup> CR risk-based indoor air response levels.

Table 3b Vapor Intrusion Matrix for Noncarcinogenic Chemicals Quanta Site, Edgewater, New Jersey

Subslab Soil Gas	Indoor Air Co	oncentrations	
Concentrations	HI < 1		Hi > 1
HI < 1	NO FURTHER ACTION. Potentially site- related and below the acceptable hazard index of     1.	Potentially site-rel hazard index. Fur identify other poter pathway(s). If non preferred option d	or PURSUE REMEDIATION ated and above the acceptabl ther investigation needed to nitial sources or preferential the found remediation is use to indoor air concentrations eptable hazard index of 1.
HI > 1	3. MONITOR or PURSUE REMEDIATION - Probably site-related and below the acceptable hazard index of 1. Additional monitoring may be advisable prior to remediation to determine the future potential for vapor intrusion from elevated soil gas concentrations. Alternatively, remediation could be performed to eliminate the potential for future exposures with potential cost savings by eliminating additional monitoring costs.	1	EDIATION - Probably site- is above the acceptable haza

Source: USEPA Region 2, 2004 HI - noncancer Hazard Index

Notes: HI < 1

HI > 1

Indoor air concentrations also will be compared with measured background concentrations (see

Section 4.3) as well as risk-based indoor air response levels based on a HI of 1.

Indoor air concentrations also will be compared with measured background concentrations (see

Section 4.4) as well as risk-based indoor air response levels based on a HI of 1.

Table 4
Response Levels for Indoor Air and Subslab Samples
Quanta Site, Edgewater, New Jersey

Constituent	CASRN			ing Levels Ba n 9 PRGs and			-							;		
				(ug/m³)				Indoor A	ir Response	Levels			Subsla	b Response	Levels	
·		10 <sup>-6</sup> risk		HQ = 1		Notes	10 <sup>-6</sup> Target Risk	10 <sup>-5</sup> Target Risk	10 <sup>-4</sup> Target Risk	HQ = 0.1	HQ =1	10 <sup>-6</sup> Target Risk	10 <sup>-5</sup> Target Risk	10 <sup>-4</sup> Target Risk	HQ = 0.1	HQ =1
	-															1
1,1-Dichloroethylene	75354			2.08E+02	N			<u> </u>			2.08E+02				2.08E+02	
1,1-Dichloroethane	75343			5.21E+02	N	<u> </u>		<u> </u>			5.21E+02		·	<u>.</u>	5.21E+02	5.21E+03
1,1,1-Trichloroethane	71556			2.30E+03	N						2.30E+03	ļ			2.30E+03	
1,1,2-Trichloroethane	79005	1.20E-01	С	1.46E+01	<u> </u>		1.20E-01	1.20E+00		1.46E+00			1.20E+01	1.20E+02	1.46E+01	1.46E+02
1,1,2,2-Tetrachloroethane	79345	3.31E-02	С	2.19E+02	<u> </u>		3.31E-02	3.31E-01	3.31E+00		2.19E+02	3.31E-01	3.31E+00	3.31E+01.		
1,2-Dibromoethane (EDB)	106934	3.40E-03	С	9.50E+00		<u> </u>	3.40E-03	3.40E-02			9.50E+00	3.40E-02	3.40E-01	3.40E+00	9.50E+00	9.50E+01
1,2-Dichloroethane	107062	7.39E-02	С	5.11E+00	$oxed{igspace}$	ļ	7.39E-02	7.39E-01	7.39E+00		5.11E+00	7.39E-01	7.39E+00	7.39E+01	5.11E+00	5.11E+01
1,2-Dichloropropane	78875	9.89E-02	С	4.17E+00			9.89E-02	9.89E-01	9.89E+00		4.17E+00	9.89E-01	9.89E+00	9.89E+01		4.17E+01
1,2,4-Trichlorobenzene	120821			3.65E+00	N	I	· ·	ļ	<u> </u>		3.65E+00		<u> </u>		3.65E+00	3.65E+01
1,2,4-Trimethylbenzene	95636			6.00E+00	N	NJDEP				6.00E-01					6.00E+00	6:00E+01
1,3-Butadiene	106990	6.11E-02	С	2.08E+00		1	6.11E-02	6.11E-01	6.11E+00		2.08E+00	6.11E-01	6.11E+00	6.11E+01	2.08E+00	2:08E+01
1,3,5-Trimethylbenzene	108678			6.00E+00	N	NJDEP		<u> </u>			6.00E+00	1	<u> </u>		6.00E+00	6.00E+01
Acetonitrile	75058			6.21E+01	N					6.21E+00	6.21E+01			,	.6.21E+01	6.21E+02
Acetylene	74862				l			L					}	· .		
Acrylonitrile	107,131	2.83E-02	С	2.09E+00			2.83E-02	2.83E-01	- 2.83E+00	2.09E-01	2.09E+00	2.83E-01	2.83E+00	2.83E+01	2.09E+00	2.09E+01
Benzene	71432	2.49E-01	С	3.14E+01	T		2.49E-01	2.49E+00	2.49E+01	3.14E+00	3.14E+01	2.49E+00	2.49E+01	2.49E+02	3.14E+01	3.14E+02
Bromochloromethane	74975				1											
Bromodichloromethane	75274	1.08E-01	С	7.30E+01	1		1.08E-01	1.08E+00	1.08E+01	7.30E+00	7.30E+01	1.08E+00	1.08E+01	1.08E+02	7.30E+01	7.30E+02
Bromoform	75252	1.75E+00	С	7.30E+01			1.75E+00	1.75E+01	1.75E+02	7.30E+00	7.30E+01	1.75E+01	1.75E+02	1.75E+03	7.30E+01	7.30E+02
Bromomethane (methyl bromide)	74839		1	5.00E+00	N	NJDEP				5.00E-01	5.00E+00				5.00E+00	5.00E+01
Carbon Tetrachloride	56235	1.28E-01	С	2.56E+00			1.28E-01	1.28E+00	1.28E+01	2.56E-01	2.56E+00	1.28E+00	1.28E+01	1.28E+02	2.56E+00	2.56E+01
Chlorobenzene	108907		1	6.20E+01	N	NJDEP	_			6.20E+00	6.20E+01				6.20E+01	6.20E+02
Chloroethane	75003		C	1.04E+04		NJDEP	2.00E+00	2.00E+0	2.00E+02			2.00E+01	2.00E+02	2.00E+03		1.04E+05
Chloroform	67663	1	С	5.11E+01	$\top$	<u> </u>	8.30E-02	8.30E-0			5.11E+01	8.30E-01	8.30E+00		5.11E+01	5.11E+02
Chloromethane (methyl chloride)	74873		İ	9.49E+01	Ī N	•		<b>—</b>		9.49E+00				-	9.49E+01	9.49E+02
Benzyl Chloride	100447	3.96E-02	c	1.06E+01	$\top$	1	3.96E-02	3.96E-0	1 3.96E+00	1.06E+00	1.06E+01	3.96E-01	3.96E+00	3.96E+01		1.06E+02
2-Chloro-1,3-butadiene	126998		1	7.30E+00	N					7.30E-01					7.30E+00	
Cis-1,2-Dichloroethylene	156592		1	3.60E+01		NJDEP					3.60E+01			-	3.60E+01	3.60E+02
Cis-1,3-Dichloropropene	10061015	<del></del>	1 c	2.09E+01	1.	1	4.80E-01	4.80E+0	4.80E+0		2.09E+01	4.80E+00	4.80E+01	4.80E+02		2.09E+02
Dibromochloromethane	124481		10	<del> </del>	1	<del> </del>	8.00E-02					8.00E-01			7.30E+01	
Dichlorodifluoromethane	75718		1	1.80E+02	IN	NJDEP					1.80E+02			·	1.80E+02	
1,2-dichloro-1,1,2,2-tetrafluoroethane	76142		+		+						1	<u> </u>				+
Ethyl Acrylate	140885		С		+	+	1.40E-01	1.40E+0	0 1.40E+0	1	1	1.40E+00	1 40F+01	1.40E+02	<del>,  </del>	<del>                                     </del>
Ethyl Tert-Butyl Ether		0	+-	<del> </del>	+	<del>- </del>	1	1	1	<del> </del>	1	1	1	1		1
Ethylbenzene	100414		+	1.06E+03	N					1.06E+02	2 1.06E+03	<del></del>	† · · · ·	<del>                                     </del>	1.06E+03	1.06E+04
Hexachloro-1,3-Butadiene	87683		c		+-`		8.62E-02	8.62E-0	1 8.62E+0		1 1.10E+00		8.62E+00	8.62E+01		
1,3-Dichlorobenzene	54173		+~	1.10E+02	N	<del>i   </del>	3.022 02	1 322 0	0.022.00		1 1.10E+02		1		1.10E+02	
Methyl ethyl ketone (2-butanone)	7,893		+	5.11E+03	1			+	<del>                                     </del>		2 5.11E+03		1	<del></del>	5.11E+03	
Methyl Isobutyl Ketone	108.10		+-	3.11E+03		NJDEP	<del></del>	<del></del>			2 3.11E+03			<del> </del>	3.10E+03	
Methyl Methacrylate	8062			7.30E+02	+		_				1 7.30E+02				7.30E+02	
Methyl tertbutyl ether (MTBE)	163404		+		+'`	NJDEP	2.00E+00	2.00E+0	1 2.00E+0			3 2.00E+01	3 00=+03	2 2.00E+0		
				3.13E+03	+	INJUER										
Methylene Chloride	7509	2 4.09E+00	<u> </u>	3.13E+03	丄		4.09E+00	4.09⊑+0	1 4.09E+0	Z 3.13E+0	∠  3.13E+03	4.09E+01	4.09E+02	4.09E+0	5   3.13E+03	3.13E+04

Table 4
Response Levels for Indoor Air and Subslab Samples
Quanta Site, Edgewater, New Jersey

Constituent	CASRN	Indoor Air So of USEPA R		n 9 PRGs and							:					
				(ug/m³)				Indoor A	ir Response I	_evels			Subsla	b Response	Levels	
		10 <sup>-6</sup> rìsk		HQ = 1		Notes	10 <sup>-6</sup> Target Risk	10 <sup>-5</sup> Target Risk	10 <sup>-4</sup> Target Risk	HQ = 0.1	HQ =1	10 <sup>-6</sup> Target Risk	10 <sup>-5</sup> Target Risk	10 <sup>-4</sup> Target Risk	HQ = 0.1	HQ =1
m-Xylene	108383			1.06E+02	N					1.06E+01	1.06E+02				1.06E+02	1.06E+03
p-Xylene	106423			1.06E+02	N			1		1.06E+01	1.06E+02				1.06E+02	1.06E+03
n-Octane	111659															
1,2-Dichlorobenzene	95501			1.50E+02	N	NJDEP				1.50E+01	1.50E+02				1.50E+02	1.50E+03
o-Xylene	95476			1.06E+02	N					1.06E+01	1.06E+02				1.06E+02	1.06E+03
1,4-Dichlorobenzene	106467	3.06E-01	С	8.40E+02			3.06E-01	3.06E+00	3.06E+01	8.40E+01	8.40E+02	3.06E+00	3.06E+01	3.06E+02	8.40E+02	8.40E+03
Propylene	115071															
Styrene	100425			1.00E+03	N	NJDEP	-			1.00E+02	1.00E+03				1.00E+03	1.00E+04
Tert-Amyl Methyl Ether	60297															
Tetrachloroethylene	127184	3.20E-01	С	3.65E+01			3.20E-01	3.20E+00	3.20E+01	3.65E+00	3.65E+01	3.20E+00	3.20E+01	3.20E+02	3.65E+01	3.65E+02
Toluene	108883			5.10E+03	N					5.10E+02	5.10E+03				5.10E+03	5.10E+04
Trans-1,2-Dichloroethylene	156605		•	6.20E+01	N	NJDEP	<u> </u>			6.20E+00	6.20E+01				6.20E+01	6.20E+02
Trans-1,3-Dichloropropene	10061026	4.80E-01	С	2.09E+01	T		4.80E-01	4.80E+00	4.80E+01	2.09E+00	2.09E+01	4.80E+00	4.80E+01	4.80E+02	2.09E+01	2.09E+02
Trichloroethylene	79016	1.68E-02	С	3.65E+01			1.68E-02	1.68Ę-01	1.68E+00	3.65E+00	3.65E+01	1.68E-01	1.68E+00	1.68E+01	3.65E+01	3.65E+02
Trichlorofluoromethane	75694			7.30E+02	N			,		7.30E+01	7.30E+02				7.30E+02	7.30E+03
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76131			3.10E+04	N	NJDEP				3.10E+03	3.10E+04				3.10E+04	3.10E+05
Vinyl Chloride	75014	1.06E-01	С	1.04E+02			1.06E-01	1.06E+00	1.06E+01	1.04E+01	1.04E+02	1.06E+00	1.06E+01	1.06E+02	1.04E+02	1.04E+03
Naphthalene	91203	3		3.13E+00	N					3.13E-01	3.13E+00				3.13E+00	3.13E+01
Notoe:																

Notes

Values shown in bold are from the NJDEP Vapor Intrusion Guidance (NJDEP, 2005; NJDEP, 2006).

Measured ambient background concentrations will be used as investigation response levels if these are higher than regulatory screening levels Soil gas response levels are calculated from indoor air levels using a default attenuation factor of 0.1

PRGs - Preliminary Remediation Goals

IASL - Indoor Air Screening Levels

USEPA - U.S. Environmental Protection Agency

NJDEP - New Jersey Department of Environmental Protection

**Table 5**NJDEP Rapid Action Levels and Health Department Notification Levels
Quanta Site, Edgewater, New Jersey

	,						Healtl	ı De	partment	
	Residentia	l Scr	eening Lev	els <sup>1</sup>	Rapid Acti	on Levels <sup>2</sup>	Notification Levels (HDNL) <sup>3</sup>			
Chemical	ug/m³		ppbv		ug/m³	ppbv	ug/m³		ppbv	
Acetone	3300	N	1400	N	6600	2800	31000	а	13000	а
Benzene	2	*C	0.5	С	200	50	80	а	25	а
Carbon Tetrachloride	3	*C	0.5	С	300	50	100		20	
Chloroform	2	*C	0.5	С	200	50	80	Γ	20	
1,2-Dichloroethene (total)	33	N	8	N	66	16	400	а	100	a
Ethylbenzene	1100	N	240	N	2200	480	4300	i	1000	Į i
Methylene chloride	4	С	1	С	400	100	1000	а	300	а
MTBE (methyl tert-butyl ether)	2	*C	0.5	С	200	50	2000		400	
Tetrachloroethylene	3	*C	0.5	С	300	50	300		50	
Toluene	5100	N	1400	N	10000	2800	>5100		>1400	
Trichloroethene	3	*C	0.5	С	20	3	20		3	
Vinyl chloride	1	*C	0.5	С	100	50	70		30	
Xylenes (total)	110	N	25	N	220	50	2200	а	500	а

Source: Table 2, "NJDEP Action Levels for Indoor Air" (NJDEP, 2006)

- 1. Residential Screening Level is the higher of the health-based value and the TO-15 analytical reporting limit. An asterik indicates the value is based on the higher analytical reporting limit. C = cancer-based value. N = noncancer-based value.
- 2. Rapid Action Levels are based on a factor of 100x for carcinogens and a factor of 2x for noncarcinogens.
- 3. Health Department Notification Levels are based on 1/2 of the Acute MRL or 1,000x the cancer risk-based residential indoor air screening level, whichever is lower. The Intermediate-Duration MRL is used in cases where Acute-Duration MRL values are not available.

The HDNL for toluene is set at exceedence of the residential screening level to reflect recent updates in the reference concentration (RfC) toxicity factor not yet incorporated in the ATSDR acute MRL value.

**Table 6**Preliminary Sampling Locations, Vapor Intrusion Investigation, 115 River Road Building Quanta Resources Site, Edgewater, NJ

Sampling Location	Indoor Air Sample	Ambient (outdoor) Air Sample	Subslab Sample	Notes
THE THE PERSON NAMED IN THE PERSON NAMED IN	115 River Road Building	, from west (Interchange Bar	rk) to east (the pier addition) is a	
Building 11 (Bank Building)	None - cannot get access over the weekend because of bank security requirements.		NA	Information will be developed regarding ventilation rates, air exchanges, HVAC system details and other building specific issues that may influence the potential for vapor intrusion at this building.
Building 10	3 samples - one first floor, one from the second OR third floor; one from the basement adjacent to the mechanical room sump.		2 samples - in Building 10 basement (sketch map with locations provided in Attachment 1)	Indoor air samples will be located in tenant spaces (offices). Specific locations will be based on building owners being able to obtain access.
Building 9 (modeling agency) .	2 samples - one first floor, one from the second OR third floor	NA	NA	Indoor air samples will be located in tenant spaces (offices). Specific locations will be based on building owners being able to obtain access.
Building 8	2 samples - one first floor, one from the second OR third floor	NA		Indoor air samples will be located in tenant spaces (offices). Specific locations will be based on building owners being able to obtain access.
Building 7 (day care center)	5 samples - one sample in each of four classrooms, and one sample from the staff room.	NA		Basement under Building 8 is also underneath Building 7 - substab samples in this location also will evaluate Building 7 conditions.  Indoor air samples will be placed at a height between 1 to 2 feet above the ground in the classrooms, to simulate breathing zone height.
Building 3	2 samples - one first floor, one from the second OR third floor		hallway, first floor	No basement in this portion of the building from Building 6 to the pier Indoor air samples will be located in tenant spaces (offices). Specific locations will be based on building owners being able to obtain access.
Pier Building	2 samples- in available tenant spaces	NA		Indoor air samples will be located in tenant spaces (offices). Specific locations will be based on building owners being able to obtain access.

Table 6

Preliminary Sampling Locations, Vapor Intrusion Investigation, 115 River Road Building

Quanta Resources Site, Edgewater, NJ

Sampling Location	Indoor Air Sample	Ambient (outdoor) Air	: Subslab Sample	Notes
		Sample	1.	
Building 12 (smaller building to the north of	2 samples - in available tenant	NA	2 samples - in the parking area next	Indoor air samples will be located in tenant
the larger 115 River Road Building)	spaces		to brick pilings	spaces (offices). Specific locations will be
				based on building owners being able to
				obtain access.

Ambient Background Sampling Locations (locations depicted in Figure 3): One sample per location

915 River Road (City of Edgewater parking lot for municipal vehicles and ambulance garage) - 1.8 mi north of the site

838 River Road (City of Edgewater Volunteer Fire Department substation) - 1.6 minorth of the site

Quanta Resources Site fenceline, between the Quanta site and former Celotex property to the north

Quanta Resources Site fenceline, next to the Hudson River

Between River Road and 115 River Road building location

#### Sample Summary

	Indoor Air	Ambient (outdoor) Air	Subslab	Total
Field Samples	18	7	9	34
Field Duplicates	2	1	11	4
Total Sample Numbers	20	8	10	38

Notes:

NA Not Applicable

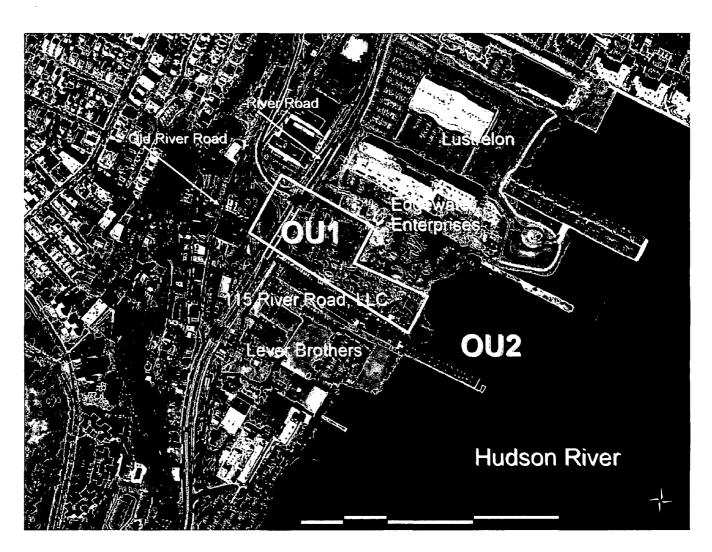
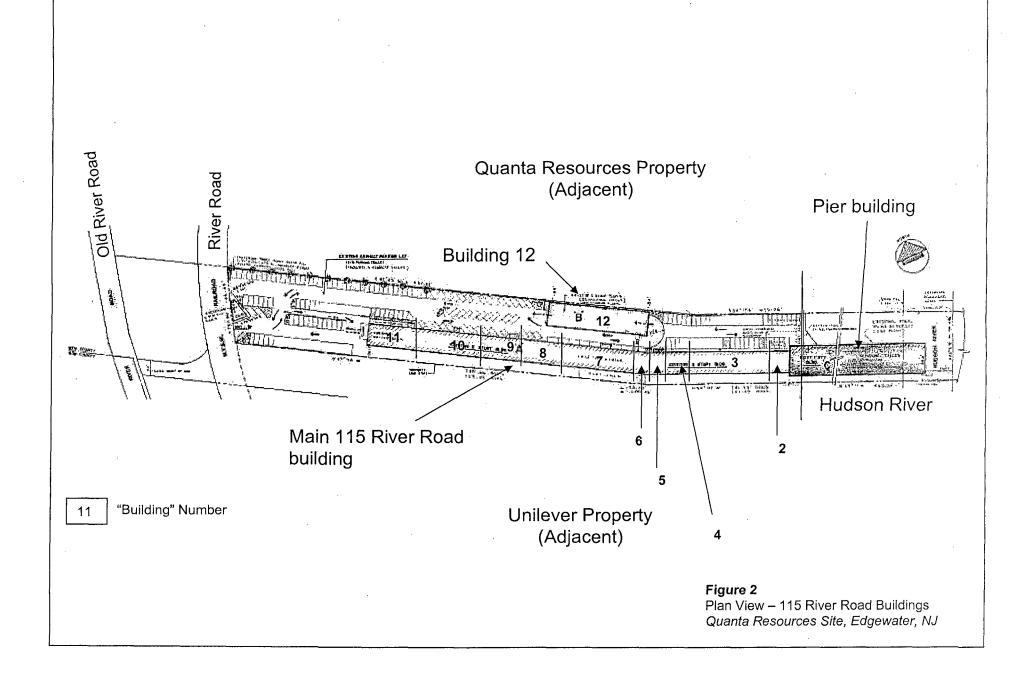


Figure 1
Location of Quanta Resources Site – Operable
Units 1 and 2, and Adjacent Properties
Quanta Resource Site, Edgewater NJ



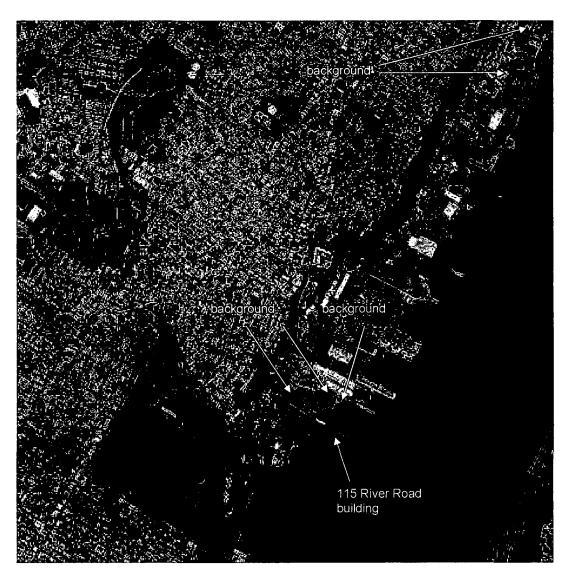


Figure 3
Proposed Background Air Sampling Locations
Quanta Resource Site, Edgewater NJ

Appendix A

Quality Assurance Project Plan Amendment

## QUALITY ASSURANCE PROJECT PLAN AMENDMENT

# OPERABLE UNIT 1, VAPOR INTRUSION EVALUATION

## QUANTA RESOURCES SITE EDGEWATER, NEW JERSEY

Prepared For:

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March 2006

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#### **ATTACHMENTS**

- U.S. Environmental Protection Agency (EPA). January 1999. Compendium Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS) from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. Second Edition.

  (http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-15r.pdf) Center for Environmental Research Information. Office of Research and Development. Cincinnati, OH.
- 2 Standard Operating Procedures
- 3 Example Building Survey Questionnaire, Example Field Data Sheet and Example Chain of Custody record

## **Project Description**

### 1.1 Problem Definition/Background

The purpose of this Quality Assurance Project Plan (QAPP) is to describe the procedures to be used for the environmental sampling on the southern portion of the Quanta Resources (Quanta) site located in Edgewater, New Jersey.

This description of the site setting was summarized from the Removal Site Investigation (RSI) report prepared by Geosyntec (2000). The Quanta property is located in Bergen County, at 163 River Road, Edgewater, New Jersey. The Quanta property is bordered to the north by the Celotex and Lustrelon Industrial Park. The former Spencer Kellogg Industrial Park is located to the south. The Hudson River borders the Quanta property to the east, and the (old) River Road borders the property to the west. The new River Road is located east of the former location, bisecting the Quanta property.

Past uses of the Quanta site include a roofind plant that manufactured creosote, coal tar pitches and refined tar for road construction, and a waste oil reprocessing plant. Current the Quanta site is vacant. The Lustrelon and northern portion of the Celotex properties have undergone redevelopment for commercial and residential uses. During redevelopment, an additional several feet of fill was imported and graded to bring the property 6 to 8 feet above the grade of the Quanta property. The Spencer Kellogg Property has been redeveloped and currently includes the Bridgeview Bank, various offices, a newly reconstructed dock containing parking and offices, and a daycare center for property tenants. South of the Spencer Kellogg property is the Lever Brothers Property, owned by Unilever. Volatile organic compounds (VOCs) detected in soil at the Quanta site principally consisted of benzene, toluene, ethylbenzene, xylenes (BTEX), and napthalene, present along the southern boundary of the site.

In order to determine the potential vapor intrusion pathway into the Spencer Kellogg building (115 River Road building), indoor and ambient (outdoor) air monitoring, subslab sampling, and soil gas sampling and analysis are needed to evaluate VOCs potentially in the subsurface in proximity to the 115 River Road building. Analytical work will be performed by Columbia Analytical Services, Simi Valley.

### 1.2 Project/Task Description

The objective of this study is to the current potential for indoor air vapor intrusion in the Spencer Kellogg building from VOCs in subsurface. This evaluation will involve comparing sample results to response levels in indoor air, and subslab screening levels. The response levels and subslab screening levels have been developed from available risk-based screening values developed by the U.S. Environmental Protection Agency (USEPA). The values are from USEPA's Integrated Risk Information System (IRIS),

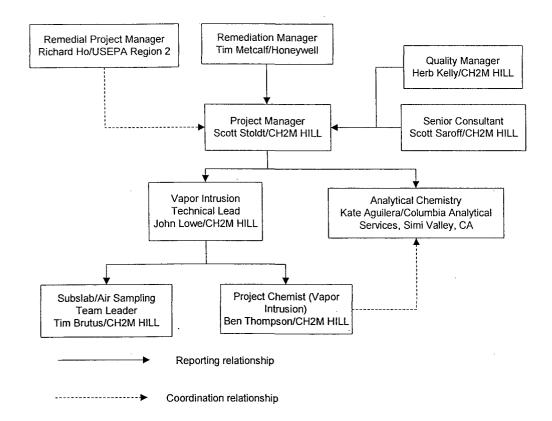
National Center for Environmental Assessment (NCEA), and/or USEPA Region 9 preliminary remediation goals (PRGs). Levels of concern for carcinogens represent a 10-6 (one in one million) to 10-4 (one in ten thousand) excess individual lifetime cancer risk. Noncancer levels of concern represent a hazard quotient ranging from 0.1 to 1. The constituent-specific concentrations corresponding to each response level are presented in Table A-1.

The purpose for the sampling and analysis data collected during this vapor intrusion evaluation will to identify potentially complete vapor intrusion pathways into the 115 River Road building, if any, and to identify appropriate actions to address potentially complete vapor intrusion pathways.

## **Project Organization and Responsibility**

## 2.1 Project/Task Organization

The following organization chart presents the key personnel and their corresponding responsibilities.



## **QA Objectives for Measurement Data (PARCC)**

## 3.1 Quality Objectives and Criteria for Measurement Data

To assess data quality, PARCC (precision, accuracy, representativeness, completeness, and comparability) parameters will be utilized. This is an integral part of the overall monitoring network design. Precision and accuracy are expressed in purely quantitative terms. The other parameters are only expressed using a mixture of quantitative and qualitative terms. All of these parameters are interrelated in terms of overall data quality and they may be difficult to evaluate separately due to these interrelationships. The relative significance of each of the parameters depends on the type and intended use of the data being collected. Therefore, these essential data quality elements are delineated as follows.

#### 3.1.1 Analytical and Sample Collection Precision

The measure of replicate precision is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

Percent difference = 
$$\frac{|X_1 - X_2|}{X} \times 100$$

where:  $X_1$  - First measurement value

X<sub>2</sub> - Second Measurement value X - Average of the two values

Factors that affected the precision of the measurement are: molecular weight, water solubility, polarizability, etc. A primary influence is the concentration level of the compound. A replicate precision value of 25 percent can be achieved for each of the target compounds. For more information, refer to Compendium Method TO-15: Determination of

VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass. Spectrometry (GC/MS), which can be found as Appendix A.

#### 3.1.2 Analytical and Sample Collection Accuracy

A measurement of analytical accuracy is the degree of agreement with audit standards. It is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage as follows:

For more information, refer to *Compendium Method TO-15*: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS, which can be found as Appendix A. As per Method TO-15, the performance criteria for audit accuracy should be within 30 percent for concentrations normally expected within contaminated ambient air.

#### 3.1.3 Data Representativeness

As previously discussed, data representativeness will be assessed by collecting field colocated samples. The field co-located samples are by definition equally representative of a given point and space and time. The laboratory also collects two samples from one canister; the sample and a duplicate. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. Therefore, data representativeness will be satisfied by ensuring that:

Proper sampling techniques are used in accordance with the SOPs enclosed in Attachment 2:

SOP 1	Collection of Sub-Slab Vapor Samples Using Summa Canisters
SOP 2	TO-14/15 (Summa Canister) Integrated Ambient or Indoor Air Sampling Method for Trace VOCs
SOP 3	TO-14/15 Flow Controller Cleaning (Purging) Method
SOP 4	TO-14/15 Integrated Sampling Device (Flow Controller) Setting Method

Proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory according to:

USEPA. January 1999. Compendium Method TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. Second Edition. Center for Environmental Research Information. Office of Research and Development. Cincinnati, OH, which can be found as Attachment 1.

#### 3.1.4 Data Completeness

Data completeness will be expressed as the percentage of valid data obtained from measurement system. For data to be considered valid, it must meet all the acceptable criteria including accuracy and precision, as well as any other criteria specified by the analytical method used. Therefore, all data points critical to the sampling program in terms of completeness will be 100 percent validated by CH2M HILL.

### 3.1.5 Data Comparability

To ensure data comparability, sampling and analysis for all samples will be performed using standardized analytical methods and adherence to the quality control procedures outlined in the methods and this QAPP.

## **Sampling Procedures**

### 4.1 Pre-Sampling Investigation

A qualitative assessment of the factors influencing indoor air contamination will be evaluated by conducting a simple "walk through" assessment prior to sampling. During this assessment observations about potential indoor sources of the particular compounds noted in Table A-1 should be made. Any other influencing factors will be noted and logged. A *Questionnaire* (see Attachment 3) will be completed during the survey. The actual number of indoor air and subslab samples for the initial sampling event will be determined following the building walkthrough survey.

## 4.2 Sampling Process Design

Whole air samples are to be collected in evacuated SUMMA canisters, and analyzed in accordance with Compendium Method TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS (USEPA 1999) from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

Following the building walkthrough survey, and USEPA acceptance of sampling locations, one round of indoor air and subslab samples will be collected from within the Spencer Kellogg building. The subslab sample probes will remain in place for follow-up sampling. USEPA will be contacted before the sample probes are abandoned. It is anticipated that the indoor air sampling event will be performed during a "cold" season, i.e. before the end of March. A second indoor air sampling event will be conducted during the warm season (i.e. between April and October). The results from these two sampling events will be evaluated to determine additional steps, if any, that may be needed to evaluate or mitigate the potential vapor intrusion pathway.

Ambient background air samples will be collected from outdoor locations beyond the footprint of affected groundwater at the Quanta site to supplement literature values for typical ambient levels of constituents in air. The results from the ambient air sampling and literature values will be used to develop background values in air. The background values in air will be used along with risk-based concentrations to evaluate indoor air sampling results (described previously under response levels) and assist in selection of constituents of interest (COIs). Ambient air sampling locations, numbers and methods for evaluating ambient air data will be described in the vapor intrusion evaluation work plan.

Additional indoor air sampling events may be conducted based on the results of the initial synoptic indoor air and subslab sampling. The frequency and locations of these samples will be determined based on comparison of air sampling results with the response levels.

Indoor air and ambient air samples will be integrated over a 24-hour period. Soil gas and sub-slab samples will be grab samples taken over a 5 minute period. In addition, colocated samples (field duplicates) will be collected by placing two canisters side by side and opening the valves simultaneously. Sample frequency should be at least one colocated sample for every 10 field samples. The proposed sampling and analysis protocol is listed in Table A-2.

TABLE A-2 Sampling and Analysis Protocols								
Sample Type	Numb er of Samp les <sup>2,3</sup>	M a t r i	Parameter/ Fraction	Sampl e Conta iner	Sample Preserva tion	Analy tical Meth od <sup>1</sup>	Method Detection Limit	Hold ing Tim e
Indoor Air & Ambient Backgrou nd	28	A i r	Volatile Organic Compound s (VOCs)	SUM MA Cani ster		TO- 15	See Table A- 3	14 day s
Sub Slab	. 11	A i r	Volatile Organic Compound s (VOCs)	SUM MA Cani ster		TO- 15	See Table A- 3	14 day s

#### Legend:

USEPA. January 1999. Compendium Method TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. Second Edition.

Performance of the follow up sampling events will be determined following evaluation of the results of the initial sampling events. The actual number of follow-up sampling events and numbers of samples per event may differ from what is proposed, depending on the results of the initial sampling event.

## 4.3 Sampling Methods Requirements

#### 4.3.1 Standard Operating Procedures

The specific standard operating procedure (SOP) utilized for air sampling are presented in Attachment 2.

### 4.3.2 Sample Collection Methodology

All samples including quality assurance/quality control (QA/QC) samples will be collected by CH2M HILL from the buildings in the area of the site.

There will be a total of 28 indoor air and ambient background samples proposed, including field quality control samples (i.e. field co-located sample). Samples will be collected by placing a 6 liter SUMMA<sup>TM</sup> canister in the appropriate location with a pre-set valve for twenty-four hour sampling.

There will be a total of eleven subslab samples proposed, including field quality control samples. Samples will be collected by placing a 850 milliliter (mL) or 1 liter SUMMA<sup>TM</sup> canister in the appropriate location with a preset valve for 5-minute sampling.

#### 4.3.3 Sample Containers, Volume, Preservation, and Holding Times

Sample container type, volume, preservation, and holding times are dependent upon analytical parameter and fraction and are matrix specific. The following table outlines the sample container type, volume, preservation, and holding times for samples to be collected onsite.

Analytical Parameter/Fraction	Sample Container	Required Sample Volume	Sample Preservation	Holding Time
VOC Indoor & Ambient	(1) SUMMA <sup>TM</sup> canister	6 liter		14 days to analyze
VOC Sub slab/soil gas	(1) SUMMA <sup>TM</sup> canister	0.85-1 liter		14 days to analyze

#### 4.3.4 Field Measurement Data Collection

Canister Sample Data Sheets, Questionnaires, Chain of Custody and the field notebook will be completed for each sample collected. The Questionnaire will record sample location; building information; time of sample drop off and pick up; conditions in the room; laboratory sample number; laboratory sample analysis and sample collection notes and/or observations. An example of the Questionnaire is presented in Attachment 3. The Field Data Sheet will be used to record the sample location, sampling period, initial and final sample time, can ID, flow controller ID, initial and final pressure and comments. An example of this data sheet can also be found in Attachment 3. The Chain of Custody is a record of the sample location, sample canister and valve numbers and time and date of the sample. An example of the Chain of Custody can be found in Attachment 3. The field notebook will be completed as provided for in Section 8.4: Data Quality Management of the QAPP.

### 4.3.5 Sampling Equipment Decontamination

Air samples will be collected using SUMMA<sup>TM</sup> canisters. The laboratory will clean and certify the canister prior to sending them to the site and will provide CH2M HILL with documentation certifying them as clean. This documentation should be included in the final report. The SUMMA<sup>TM</sup> canisters will be cleaned and certified according to TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (USEPA 1999)

### 4.3.6 Management of Investigative-Derived Wastes (IDW)

There will be minimum waste due to the nature of the sampling event. There will be no personnel protective equipment to be disposed.

## Sample Custody

### 5.1 Special Training Requirements/Certification

To perform the operations of this sampling event, sampling personnel may be dealing with remedial or removal activities onsite. This could expose sampling personnel to potential occupational environmental hazards. As a result, it is important for field personnel to be familiar with:

- Identifying methods and procedures for recognizing, evaluating and controlling hazardous substances.
- Identifying concepts, principles, and guidelines to properly protect field personnel.
- Discussing regulations and action levels to ensure the health and safety of field oversight personnel.
- Discussing the fundamentals needed to develop organizational structures and standard operating procedures to mitigate potential environmental hazards.
- Demonstrating the selection and use of dermal and respiratory protective equipment.
- Demonstrating the selection and use of direct-reading air monitoring instrumentation (if applicable)
- Review of any site-specific Health and Safety Plans

In practice, not all of the potential environmental hazards which may be inherent to a site can be readily anticipated. To mitigate these circumstances, field personnel must learn, follow, and enforce the published rules governing occupational health and safety. In addition, they must maintain awareness and exercise common sense and good judgment when confronting possible unsafe situations.

### 5.2 Sample Handling and Custody Requirements

#### 5.2.1 Sample Handling and Shipment

Field Data Sheets, Chain of Custody and the field notebook will be completed for each sample collected. All field and sample documents will be legibly written in indelible ink. Any corrections or revisions will be made by lining through the original entry and initialing the change. The Field Data Sheet will be provided by the project and records the sample location, sampling period, initial and final sample time, comments and other pertinent sampling information. An example of this data sheet can also be found in Attachment 3. The Chain of Custody is a record of the sample location, sample canister and valve numbers and time and date of the sample. An example of the Chain of Custody can be found in Attachment 3.

The field notebook will be used by field personnel to record all aspects of sample collection

and handling, visual observations, and field measurements. The field notebook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. The sample team or individuals performing a particular sampling activity are required to maintain a field notebook. This field notebook will be a bound weatherproof logbook that shall be filled out at the location of sample collection immediately after sampling. All entries will be signed by the individuals making them. At a minimum, the notebook will contain sample particulars including sample number, collection time, location, descriptions, methods used, daily weather conditions, field measurements, name of sampler(s), sample preservation, names of contractor/subcontractor personnel, and other site-specific observations including any deviations from protocol.

A canister tag, also provided by the laboratory, will be securely affixed to each SUMMA<sup>TM</sup> canister and include the sample ID, sampling date and time, canister identification number, and the flow controller number and flow rate. The sample tags will be secured to the canister itself. Once sealed, samples will be placed back into the cardboard boxes that they were received in. Custody seals and strapping tape will then be affixed to the boxes.

Samples will be packaged and shipped in accordance with USEPA, Department of Transportation (DOT), and International Air Transport Association (IATA) procedures. All samples will be shipped within 24 hours of collection to the laboratory.

#### 5.2.2 Sample Custody Procedures

The Chain of Custody records will be maintained from the time of sample collection until final disposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept for each individual who has signed it. The Chain of Custody records will include, at a minimum, sample identification number, number of samples collected, sample collection date and time, sample type, sample matrix, sample container type, sample analysis requested, sample preservation, and the name(s) and signature(s) of samplers and all individuals who have had custody. An example can be found in Attachment 3. Canister tags will include the canister identification number and the valve number and flow rate. Custody seals will demonstrate that a sample container or cooler has not been opened or tampered with. The sampler will sign and date the custody seal and affix it to the container and/or cooler in such a manner that it cannot be opened without breaking the seal.

## **Calibration Procedures and Frequency**

## 6.1 Instrument Calibration and Frequency

Laboratory analytical equipment calibration will follow procedures as specified under Compendium Method TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (USEPA 1999), which can be found as Attachment 1. Additional QC requirements may be found in Region 2 Data Validation SOPs available at: http://www.epa.gov/region02/qa/documents.htm.

## **Analytical Procedures**

## 7.1 Analytical Methods Requirements

The analytical method, equipment and method performance requirements for analysis will be according to TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (USEPA 1999), which can be found as Attachment 1. Additional QC requirements may be found in Region 2 Data Validation SOPs available at: http://www.epa.gov/region02/qa/documents.htm.

The reporting limits and minimum detection limits using Method TO-15 for this project are presented in Table A-3.

## Data Reduction, Validation, and Reporting

### 8.1 Data Review, Validation and Verification Requirements:

Standard methods and references will be used as guidelines for data reduction and reporting. All data generated by the laboratory will be reported in standard deliverable format and accompanied by an electronic data deliverable.

#### 8.2 Validation and Verification Methods

Analytical data will be reviewed by the Project Chemist to determine if the samples were analyzed in according to the project requirements and to assess the potential impact of laboratory performance or matrix effects on the validity of the analytical results. One hundred percent of the data will undergo a data summary validation using a combination of electronic and manual review using the applicable quality criteria in the *National Functional Guidelines for Organic Data Review* (EPA, February 1994) and Region 2 Data Validation SOPs available at: http://www.epa.gov/region02/qa/documents.htm.

The data review will include a review of the following items:

- Chain of custody and completeness of reports
- Sample receipt conditions
- Case narrative information
- Holding times
- Laboratory control sample accuracy
- Method blank results
- Field and laboratory duplicate precision
- Surrogate recovery accuracy
- Internal standard recovery accuracy and retention time precision
- Initial and continuing calibration verification precision and accuracy
- GC/MS instrument tune criteria

## 8.3 Data Acquisition Requirements

Data acquisition from nondirect measurements such as data from databases or literature is not anticipated at this time; therefore, this is not applicable.

## 8.4 Data Quality Management

All project data and information must be documented in a format that is usable by project personnel. This section of the QAPP describes how project data and information will be documented, tracked, and managed from their generation in the field to final use and storage in a manner that ensures data integrity and defensibility. All field and

sample documents will be legibly written in indelible ink. Any correction or revisions will be made by lining through the original entry and initialing the change.

The following field and sample documentation will be maintained:

- The field notebook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. The sample team or individuals performing a particular sampling activity are required to maintain a field notebook. This field notebook will be a bound weatherproof logbook that shall be filled out at the location of sample collection immediately after sampling. All entries will be signed by the individuals making them. At a minimum, the notebook will contain sample particulars including sample number, collection time, location, descriptions, methods used, daily weather conditions, field measurements, name of sampler(s), sample preservation, and other site-specific observations including any deviations from protocol.
- Field data sheets, i.e., Questionnaire, Field Data Sheet, and corresponding sample labels are used to identify samples and document field sampling conditions and activities. The field data sheets will be completed at the time of sample collection and will include the following: sample location; sample ID; building information; sample collection start and stop time and date; sample environment description; canister and flow controller number; pre and post canister pressure and sample collection notes and/or observations. An example of the Questionnaire and the Field Data Sheet are presented in Attachment 3. Sample labels will be securely affixed to the sample container and include only the sample identification number as per protocol.
- Canister tags will be securely affixed to the SUMMA<sup>TM</sup> canister and include the canister identification number and the valve number and flow rate. The tags will be fixed to the top of the canisters to prevent sample identification problems.
- The *Chain of Custody* records will be maintained from the time of sample collection until final disposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept for each individual who has signed it. The chain-of-custody records will include, at a minimum, sample identification number, number of samples collected, sample collection date and time, sample type, sample matrix, sample container type, sample analysis requested, sample preservation, and the name(s) and signature(s) of samplers and all individuals who have had custody. An example of the chain of custody that will be used at this site can be found in Attachment 3.
- Custody seals will demonstrate that a sample canister or box has not been opened or tampered with. The sampler will sign and date the custody seal and affix it to the box in such a manner that it cannot be opened without breaking the seal.
- Procedures are provided for project personnel to make changes, take corrective actions and document the process through Corrective Action Request Forms. Corrective action can occur during field activities, laboratory analysis, data validation, and data assessment. For further information, refer to Section 13, Corrective Action.

## Internal Quality Control Checks and Frequency

### 9.1 Quality Control Requirements

As previously stated, to assess data quality, PARCC parameters will be utilized. These essential data quality elements are delineated as follows.

#### 9.1.1 Data Precision

Precision is defined as a measure of the reproducibility of individual measurements of the same property under a given set of conditions. The overall precision of measurement data is a mixture of sampling and analytical factors.

#### **Analytical Precision**

The measure of replicate precision is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

Percent difference = 
$$\frac{|X_1 - X_2|}{X} \times 100$$

where:  $X_1$  - First measurement value

X<sub>2</sub> - Second Measurement value

X - Average of the two values

Factors that affected the precision of the measurement are: molecular weight, water solubility, polarizability, etc. A primary influence is the concentration level of the compound. The replicate precision value should be ≤25 percent for each of the target compounds detected above the reporting limit (see Table 4). For more information, refer to Compendium Method *TO-15*: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS (USEPA 1999), which can be found as Attachment 1.

#### Sample Collection Precision

Sample collection precision will be assessed by collecting field co-located, or field duplicate, samples. The field co-located samples will be used to evaluate errors associated with sample heterogeneity, sampling methodology, and analytical procedures. The analytical results from these samples will provide data on the overall measurement precision. The field duplicate precision value should be  $\leq$ 25 percent for each of the target compounds detected above the reporting limit (see Tables A-3 and A-4).

#### 9.1.2 Data Accuracy

Accuracy is defined as the degree of difference between measured or calculated values and the true value. The closer the numerical value of the measurement comes to the true value, or actual concentration, the more accurate the measurement is. It is difficult to

measure accuracy for the entire data collection activity. Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation and analysis techniques.

## **Analytical Accuracy**

A measurement of analytical accuracy is the degree of agreement with laboratory control spike (LCS). It is defined as the difference between the true concentration of the compound and the measured value divided by the true value and expressed as a percentage as follows:

Accuracy, % = Spiked Value - Observed Value X 100 Spiked Value

For more information, refer to Compendium Method TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS (USEPA 1999), which can be found as Attachment 1. The percent accuracy should be ≤30 percent for each of the target compounds (see Table 4).

## Sample Canister and Analysis Integrity

Method blanks will be used to monitor possible laboratory contamination. The laboratory shall provide results for the method blanks or the blank analysis for the canisters prior to use.

# 9.1.3 Data Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or and environmental condition. For the purposes of this QAPP, data representativeness is the minimum number of samples that are needed to evaluate the indoor air environment. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program and proper laboratory protocol. The representativeness criterion is best satisfied by making certain that sampling locations are selected properly and a sufficient number of samples are collected. Therefore, data representativeness will be assessed by collecting field replicate samples. The field replicates are by definition equally representative of a given point in space and time. The laboratory also collects two samples from one canister; the sample and a duplicate. In addition, as previously stated, data representativeness will be satisfied by ensuring that the sampling program is followed. Also, proper sampling techniques will be used in accordance with the SOPs in Attachment 2.

# 9.1.4 Data Comparability

Comparability is defined as the confidence with which one data set can be compared to another. Field and laboratory procedures greatly affect comparability. Therefore, to optimize comparability, sampling and analysis for all samples will be performed using standardized analytical methods and adherence to the quality control procedures outlined in the methods and this QAPP.

# 9.1.5 Data Completeness

Completeness is defined as the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data completeness will be expressed as the percentage of valid data obtained from measurement system. For data to be considered valid, it must meet all the acceptable criteria including accuracy and precision, as well as any other criteria specified by the analytical method used. Therefore, all data points critical to the sampling program in terms of completeness will be validated by CH2M HILL. The completeness goal for this sampling project will be 90 percent, although sample results significantly outside quality control criteria may be re-collected based on project decisions.

# **Preventive Maintenance**

# 10.1 Instrument/Equipment Testing, Procedures & Scheduled Inspection and Maintenance Requirements

As previously stated, calibration and preventative maintenance of analytical laboratory equipment will be conducted by the laboratory. The laboratory will also be responsible for calibration and preventative maintenance of the field sampling equipment including: sampling canisters, flow controllers, and gauges.

# 10.2 Inspection/Acceptance Requirements for Supplies and Consumables

Due to the nature of air sampling rinsate and trip blanks are not applicable. SUMMA<sup>TM</sup> canister quality control includes certification of the canister, method blanks performed by the laboratory and laboratory control samples, also performed by the laboratory. Canisters will be cleaned and certified to the limit specified in method TO-15.

**SECTION 11** 

# Specific Routine Procedures/measurement Parameters Involved

# 11.1 Reconciliation with Data Used to Assess PARCC for Quality Objectives Measurement

Sample collection precision will be evaluated by collecting and analyzing a co-located sample. The co-located samples will be used to evaluate errors associated with sample heterogeneity, sampling methodology and analytical procedures. The analytical results from the co-located sample will provide data on the overall measurement precision. Precision will be reported as the relative percent difference (RPD) for two measurements. The acceptance criteria for the co-located samples are located in Table 4, below. The laboratory also collects two samples from one canister; the sample and a duplicate.

Data will be generated through the collection of air samples in appropriate locations (e.g., indoor air, soil gas) in the area of the Site. This data will be used to determine the location and concentration of contamination in the structure, the extent of contamination, evaluate potential health threats, and determine environmental impacts while identifying clean-up criteria.

TABLE A-4: PRECISION A	ND ACCURA	<b>ACY</b>						
Sample Parameter/ Fraction	Sample Matrix	Analytical Method	Method Detection Limit <sup>1</sup>	Quantitation Limit	Estimated Accuracy <sup>2</sup>	Accuracy Protocol	Estimated Precision <sup>2</sup>	Precision Protocol
Volatile Organic Compounds (VOCs)	Air	TO-15	See Table A-3	ppbv	≤ 30%	Non-RAS	≤ 25%	Non-RAS

<sup>&</sup>lt;sup>1</sup> The method detection limits were provided by the laboratory.

<sup>&</sup>lt;sup>2</sup> TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS

# **Corrective Action**

# 12.1 Assessments and Response Actions

Procedures are provided for project personnel to make changes, take corrective actions and document the process through Corrective Action Request Forms. Corrective action can occur during field activities, laboratory analysis, data validation, and data assessment.

Corrective action in the field may be necessary when the monitoring network design is changed. A change in the field includes: increasing the number or type of samples or analyses; changing sampling locations; and/or modifying sampling protocol. When this occurs, the project manager or project chemist will identify any suspected technical or QA deficiencies and note them in the field logbook. The project chemist will be responsible for assessing the suspected deficiency and determining the impact on the quality of the data. Development of the appropriate corrective action will be the responsibility of the RPM.

Data validation and data assessment corrective action will be in accordance with the National Functional Guidelines for Organic Data Review (USEPA, February 1994).

**Table A-1**Response Levels for Indoor Air and Subslab Samples *Quanta Site, Edgewater, New Jersey* 

Constituent	CASRN			ning Levels I on 9 PRGs ar (ug/m³)				Indoor A	ir Response	l avals			Subsi	ıb Response	e l evels	
				(09/11/		1	10 <sup>-6</sup> Target	10.5 Target		Levels		10 <sup>-6</sup> Target	10 <sup>-5</sup> Target			
		10 <sup>-6</sup> risk	:	HQ = 1		Notes	Risk	Risk	Risk	HQ = 0.1	HQ ≈1	Risk	Risk	Risk	HQ = 0.1	HQ =1
												ŀ				
1,1-Dichloroethylene	75354		1	2.08E+02	N	1				2.08E+01	2.08E+02				2.08E+02	2.08E+03
1,1-Dichloroethane	75343		1	5.21E+02	N			· · · · · · · · · · · · · · · · · · ·			5.21E+02	<del> </del>	T		5.21E+02	5.21E+03
1.1.1-Trichloroethane	71556		+	2.30E+03	N					2.30E+02			<del> </del>	<del> </del>	2,30E+03	2.30E+04
1,1,2-Trichloroethane	79005	1.20E-01	c	1.46E+01	+"	<del> </del>	1.20E-01	1.20E+00	1.20E+01	1.46E+00		1.20E+00	1.20E+01	1.20E+02	1.46E+01	1.46E+02
1,1,2,2-Tetrachloroethane	79345		<del>1 č</del>	2.19E+02	+	<del> </del>	3.31E-02	3.31E-01	3,31E+00	2.19E+01		3.31E-01	3.31E+00	3.31E+01	2,19E+02	2.19E+03
1,2-Dibromoethane (EDB)	106934	3.40E-03	Ť	9.50E+00	+	<del> </del>	3.40E-03	3.40E-02	3.40E-01	9.50E-01		3.40E-02	3.40E-01	3,40E+00	9.50E+00	9.50E+01
1,2-Dichloroethane	107062	7.39E-02	č	5.11E+00	+-	<del> </del>	7.39E-02	7.39E-01	7.39E+00	5.11E-01		7.39E-01	7.39E+00	7.39E+01	5.11E+00	5.11E+01
1,2-Dichloropropane	78875	9.89E-02	Ιč	4.17E+00	+	<del> </del>	9.89E-02	9.89E-01	9.89E+00	4.17E-01		9.89E-01	9,89E+00	9.89E+01	4.17E+00	4.17E+01
1,2,4-Trichlorobenzene	120821	0.002.02	╁	3.65E+00	ĺΝ		0.002.02	0.002 01	0.002 00	3.65E-01				0.3.2.2	3.65E+00	
1,2,4-Trimethylbenzene	95636		<del>                                     </del>	6.00E+00		NJDEP	<del>                                     </del>			6.00E-01		1	<del> </del>	<del>                                     </del>	6,00E+00	6.00E+01
1.3-Butadiene	106990	6.11E-02	C	2.08E+00	1	1.35	6.11E-02	6.11E-01	6.11E+00		2.08E+00	6.11E-01	6.11E+00	6.11E+01	2.08E+00	2.08E+01
1,3,5-Trimethylbenzene	108678	0	Ť	6.00E+00	TN	NJDEP	0			6,00E-01		<del> </del>			6.00E+00	6.00E+01
Acetonitrile	75058		1	6.21E+01	T N					6.21E+00			<b></b>		6.21E+01	6.21E+02
Acetylene	74862		_	0.2.12.0	<del></del>					0,010						
Acrylonitrile	107131	2.83E-02	c	2.09E+00	+-	<del> </del>	2.83E-02	2.83E-01	2.83E+00	2.09E-01	2.09E+00	2.83E-01	2.83E+00	2.83E+01	2.09E+00	2.09E+01
Benzene	71432	2.49E-01	Ċ	3.14E+01	+	<del> </del>	2.49E-01	2.49E+00	2.49E+01	3.14E+00	3.14E+01	2.49E+00	2.49E+01	2.49E+02	3.14E+01	3.14E+02
Bromochloromethane	74975		<u> </u>		+	<del> </del>										
Bromodichtoromethane	75274	1.08E-01	С	7.30E+01	+-	<del> </del>	1.08E-01	1.08E+00	1.08E+01	7.30E+00	7.30E+01	1.08E+00	1.08E+01	1.08E+02	7,30E+01	7.30E+02
Bromoform	75252	1.75E+00	c	7.30E+01	+	<del> </del>	1.75E+00	1.75E+01	1.75E+02	7.30E+00	7.30E+01	1.75E+01	1.75E+02	1.75E+03	7,30E+01	7.30E+02
Bromomethane (methyl bromide)	74839		Ť	5.00E+00	1	NJDEP	7.1.00			5,00E-01	5,00E+00				5.00E+00	5.00E+01
Carbon Tetrachloride	56235	1.28E-01	С	2.56E+00	+ -		1.28E-01	1,28E+00	1.28E+01	2.56E-01	2.56E+00	1.28E+00	1.28E+01	1.28E+02	2.56E+00	2.56E+01
Chlorobenzene	108907		-	6.20E+01	· N	NJDEP				6.20E+00		1			6.20E+01	6.20E+02
Chloroethane	75003	2.00E+00	c	1,04E+04	+~	NJDEP	2.00E+00	2.00E+01	2.00E+02	1.04E+03		2.00E+01	2.00E+02	2.00E+03	1.04E+04	1.04E+05
Chloroform	67663	8.30E-02	č	5,11E+01	╁	1	8.30E-02	8.30E-01	8.30E+00	5.11E+00	5.11E+01	8.30E-01	8.30E+00	8.30E+01	5.11E+01	5.11E+02
Chloromethane (methyl chloride)	74873	0.002 02	۳	9.49E+01	N	i	5.502.52	0.002 01	0.002.00	9.49E+00	9.49E+01	0.002.01	0.000		9.49E+01	9.49E+02
Benzyl Chloride	100447	3.96E-02		1.06E+01	<del>  ``</del>	<del> </del>	3.96E-02	3,96E-01	3.96E+00	1.06E+00	1.06E+01	3.96E-01	3.96E+00	3.96E+01	1.06E+01	1.06E+02
2-Chloro-1,3-butadiene	126998	0.000	Ť	7.30E+00	lп	<del> </del>	0.002	5,552 51		7.30E-01	7.30E+00		3,111		7.30E+00	7.30E+01
Cis-1,2-Dichloroethylene	156592		_	3,60E+01		NJDEP				3.60E+00	3.60E+01				3.60E+01	3.60E+02
Cis-1,3-Dichloropropene	10061015	4.80E-01	С	2.09E+01	+		4.80E-01	4.80E+00	4.80E+01	2.09E+00		4.80E+00	4.80E+01	4.80E+02	2.09E+01	2.09E+02
Dibromochloromethane	124481	8.00E-02	č	7.30E+01		<del> </del>	8.00E-02	8.00E-01	8.00E+00	7.30E+00		8.00E-01	8.00E+00	8.00E+01	7.30E+01	7.30E+02
Dichlorodifluoromethane	75718			1.80E+02	N	NJDEP				1.80E+01	1.80E+02				1.80E+02	1.80E+03
1,2-dichloro-1,1,2,2-tetrafluoroethane	76142	···			1	†~ <del>~~</del>								•		
Ethyl Acrylate	140885	1.40E-01	c		1		1.40E-01	1.40E+00	1.40E+01			1.40E+00	1.40E+01	1.40E+02		
Ethyl Tert-Butyl Ether	0															
Ethylbenzene	100414			1.06E+03	N		-			1.06E+02	1.06E+03				1.06E+03	1.06E+04
Hexachloro-1,3-Butadiene	87683	8.62E-02	Ĉ	1.10E+00	1		8.62E-02	8.62E-01	8.62E+00	1.10E-01	1.10E+00	8.62E-01	8.62E+00	8.62E+01	1.10E+00	1.10E+01
1,3-Dichlorobenzene	541731			1.10E+02	N					1.10E+01	1.10E+02				1.10E+02	1.10E+03
Methyl ethyl ketone (2-butanone)	78933			5.11E+03	N					5.11E+02	5.11E+03				5.11E+03	5.11E+04
Methyl Isobutyl Ketone	108101		$\neg$	3.10E+03	N	NJDEP				3.10E+02	3.10E+03				3.10E+03	3.10E+04
Methyl Methacrylate	80626			7.30E+02	N					7.30E+01	7.30E+02				7.30E+02	7.30E+03
Methyl tertbutyl ether (MTBE)	1634044	2.00E+00	С	3.13E+03		NJDEP	2.00E+00	2.00E+01	2.00E+02	3.13E+02	3.13E+03	2.00E+01	2.00E+02	2.00E+03	3,13E+03	3.13E+04
Methylene Chloride	75092	4.09E+00	С	3.13E+03			4.09E+00	4.09E+01	4.09E+02	3.13E+02	3.13E+03	4.09E+01	4.09E+02	4.09E+03	3.13E+03	3.13E+04
m-Xylene	108383			1.06E+02	N					1.06E+01	1.06E+02				1.06E+02	1.06E+03
p-Xylene	106423			1,06E+02	N			4-1		1.06E+01	1.06E+02				1.06E+02	1.06E+03
n-Octane	111659				1	· · · · · · · · · · · · · · · · · · ·										
1,2-Dichlorobenzene	95501		_	1.50E+02	N	NJDEP				1.50E+01	1,50E+02				1.50E+02	1.50E+03
o-Xylene	95476			1.06E+02	N					1.06E+01	1.06E+02				1.06E+02	1.06E+03

Table A-1 Response Levels for Indoor Air and Subslab Samples Quanta Site, Edgewater, New Jersey

Constituent	CASRN		Indoor Air Screening Levels Based on Lowest of USEPA Region 9 PRGs and NJDEP IASLS (ug/m²)				Indoor A	ir Response	Levels			Subs	lab Response	Levels		
		10 <sup>-6</sup> risk		HQ = 1		Notes	10 <sup>-6</sup> Target Risk	10 <sup>-5</sup> Target Risk	10 <sup>-1</sup> Target Risk	HQ = 0.1	HQ ≈1	10 <sup>-8</sup> Target Risk	10 <sup>-5</sup> Targe Risk	at 10 <sup>-4</sup> Target Risk	HQ = 0.1	HQ =1
1,4-Dichlorobenzene	106467	3.06E-01	С	8.40E+02	Т		3.06E-01	3.06E+00	3.06E+01	8.40E+01	8.40E+02	3.06E+00	3.06E+01	3.06E+02	8.40E+02	8.40E+03
Propylene	115071															
Styrene	100425			1.00E+03	N	NJDEP		,		1.00E+02	1.00E+03			7	1.00E+03	1.00E+04
Tert-Amyl Methyl Ether	60297				1							T				
Tetrachloroethylene	127184	3.20E-01	С	3.65E+01			3.20E-01	3.20E+00	3.20E+01	3.65E+00	3.65E+01	3.20E+00	3.20E+01	3.20E+02	3.65E+01	3.65E+02
Toluene	108883			5.10E+03	N					5.10E+02	5.10E+03				5.10E+03	5.10E+04
Trans-1,2-Dichloroethylene	156605			6.20E+01	N	NJDEP				6.20E+00	6.20E+01				6.20E+01	6.20E+02
Trans-1,3-Dichloropropene	10061026	4.80E-01	C	2.09E+01			4.80E-01	4.80E+00	4.80E+01	2.09E+00	2.09E+01	4.80E+00	4.80E+01	4.80E+02	2.09E+01	2.09E+02
Trichloroethylene	79016	1.68E-02	С	3.65E+01			1.68E-02	1.68E-01	1.68E+00	3.65E+00	3.65E+01	1.68E-01	1.68E+00	1.68E+01	3.65E+01	3.65E+02
Trichlorofluoromethane	75694			7.30E+02	N					7.30E+01	7.30E+02				7.30E+02	7.30E+03
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76131			3.10E+04	N	NJDEP				3.10E+03	3.10E+04				3.10E+04	3.10E+05
Vinyl Chloride	75014	1.06E-01	С	1.04E+02			1.06E-01	1.06E+00	1.06E+01	1.04E+01	1.04E+02	1.06E+00	1.06E+01	1.06E+02	1.04E+02	1.04E+03
Naphthalene	91203			3.13E+00	N					3.13E-01	3.13E+00				3.13E+00	3.13E+01

Notes:

Values shown in bold are from the NJDEP Vapor Intrusion Guidance (NJDEP, 2005; NJDEP, 2006).

Measured ambient background concentrations will be used as investigation response levels if these are higher than regulatory screening levels. Soil gas response levels are calculated from indoor air levels using a default attenuation factor of 0.1

PRGs - Preliminary Remediation Goals

IASL - Indoor Air Screening Levels

USEPA - U.S. Environmental Protection Agency

NJDEP - New Jersey Department of Environmental Protection

**TABLE A-3**Minimum Reporting Limits
Quanta Site, Edgewater, New Jersey

Constituent	CASNR	Lowest risk- based value	Reporting Limit with SCAN Mode	Reporting Limit with SIM Mode
		ug/m³	ug/m³	ug/m³
1,1-Dichloroethylene	75354	21	0.1	0.025
1,1-Dichloroethane	75343	52	0.1	0.025
1,1,1-Trichloroethane	71556	230	0.1	0.025
1,1,2-Trichloroethane	79005	0.12	0.1	0.025
1,1,2,2-Tetrachloroethane	79345	0.03	0.1	0.025
1,2-Dibromoethane (EDB)	106934	0.0034	0.1	0.025
1,2-Dichloroethane	107062	0.07	0.1	0.025
1,2-Dichloropropane	78875	0.10	0.1	0.025
1,2,4-Trichlorobenzene	120821	0.37	0.1	n/a
1,2,4-Trimethylbenzene	95636	0.6	0.1	n/a
1,3-Butadiene	106990	0.06	0.1	n/a
1,3,5-Trimethylbenzene	108678	0.6	0.1	n/a
Acetonitrile	75058	6	0.1	n/a
Acetylene	74862		n/a	na/
Acrylonitrile	107131	0.03	0.1	n/a
Benzene	71432	0.2	0.1	0.1
Bromochloromethane	74975		n/a	na/
Bromodichloromethane	75274	0.1	0.1	n/a
Bromoform	75252	2	0.1	n/a
Bromomethane (methyl bromide)	74839	1	0.1	n/a
Carbon Tetrachloride	56235	0.1	0.1	0.025
Chlorobenzene	108907	6	0.1	0:025
Chloroethane	75003	2	0.1	0.025
Chloroform	67663	0.08	0.1	0.1
Chloromethane (methyl chloride)	74873	9	0.1	0.025
Benzyl Chloride	100447	0.04	0.1	n/a
2-Chloro-1,3-butadiene	126998	1	, n/a	n/a
Cis-1,2-Dichloroethylene	156592	4	0.1	0.025
Cis-1,3-Dichloropropene	10061015	0.5	0.1	0.025
Dibromochloromethane	124481	0.08	0.1	n/a
Dichlorodifluoromethane	75718	21	0.1	0.025
1,2-dichloro-1,1,2,2-tetrafluoroethane	76142		0.1	0.025
Ethyl Acrylate	140885	0.14	n/a	na/
Ethyl Tert-Butyl Ether	0		0.1	n/a
Ethylbenzene	100414	106	0.1	0.1
Hexachloro-1,3-Butadiene	87683	0.09	0.1	0.025
1,3-Dichlorobenzene	541731	11	0.1	n/a
Methyl ethyl ketone (2-butanone)	78933	511	0.5	n/a
Methyl Isobutyl Ketone	108101	314	0.1	n/a
Methyl Methacrylate	80626	73	0.1	n/a
Methyl tertbutyl ether (MTBE)	1634044	4	0.1	0.025
Methylene Chloride	75092	4	0.5	0.020
m-Xylene	108383	11	0.5	0.1
p-Xylene	106423	11	0.1	0.1
n-Octane	111659		0.1	n/a
1,2-Dichlorobenzene	95501	21	0.1	n/a
o-Xylene	95476	11	0.1	0.1

**TABLE A-3**Minimum Reporting Limits
Quanta Site, Edgewater, New Jersey

Constituent	CASNR	Lowest risk- based value	Reporting Limit with SCAN Mode	Reporting Limit with SIM Mode
1,4-Dichlorobenzene	106467	0.3	0.1	0.025
Propylene	115071		0.1	n/a
Styrene	100425	106	0.1	n/a
Tert-Amyl Methyl Ether	60297	-	0.1	n/a
Tetrachloroethylene	127184	0.3	0.1	0.025
Toluene	108883	510	0.1	0.1
Trans-1,2-Dichloroethylene	156605	7	0.1	0.025
Trans-1,3-Dichloropropene	10061026	0.5	0.1	n/a
Trichloroethylene	79016	0.017	0.1	0.025
Trichlorofluoromethane	75694	73	0.1	n/a
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76131	3128	0.1	0.025
Vinyl Chloride	75014	0.1	0.1	0.025
Naphthalene	91203	0.3	0.1	0.1

#### Notes:

NA= Not applicable - Analysis for this analyte is not feasible by this method

Analytes not addressed by this method:

Actylene - does not trap well and has poor instrument response

Bromochloromethane - is the internal standard for the method.

2-Chloro-1,3-butadiene (chloroprene) - will be addressed as a tenatively identified compound

Ethyl Acrylate - will be addressed as a tenatively identified compound

Attachment 1 USEPA TO-15: Determination of VOCs in Air Collected in Specialty-Prepared Canisters and Analyzed by GC/MS

# Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

**Second Edition** 

**Compendium Method TO-15** 

Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

Center for Environmental Research Information
Office of Research and Development
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#### DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

# **METHOD TO-15**

# Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

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#### **METHOD TO-15**

# Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

#### 1. Scope

1.1 This method documents sampling and analytical procedures for the measurement of subsets of the 97 volatile organic compounds (VOCs) that are included in the 189 hazardous air pollutants (HAPs) listed in Title III of the Clean Air Act Amendments of 1990. VOCs are defined here as organic compounds having a vapor pressure greater than 10<sup>-1</sup> Torr at 25°C and 760 mm Hg. Table 1 is the list of the target VOCs along with their CAS number, boiling point, vapor pressure and an indication of their membership in both the list of VOCs covered by Compendium Method TO-14A (1) and the list of VOCs in EPA's Contract Laboratory Program (CLP) document entitled: Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites (2).

Many of these compounds have been tested for stability in concentration when stored in specially-prepared canisters (see Section 8) under conditions typical of those encountered in routine ambient air analysis. The stability of these compounds under all possible conditions is not known. However, a model to predict compound losses due to physical adsorption of VOCs on canister walls and to dissolution of VOCs in water condensed in the canisters has been developed (3). Losses due to physical adsorption require only the establishment of equilibrium between the condensed and gas phases and are generally considered short term losses, (i.e., losses occurring over minutes to hours). Losses due to chemical reactions of the VOCs with cocollected ozone or other gas phase species also account for some short term losses. Chemical reactions between VOCs and substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long term losses over days to weeks). Loss mechanisms such as aqueous hydrolysis and biological degradation (4) also exist. No models are currently known to be available to estimate and characterize all these potential losses, although a number of experimental observations are referenced in Section 8. Some of the VOCs listed in Title III have short atmospheric lifetimes and may not be present except near sources.

- 1.2 This method applies to ambient concentrations of VOCs above 0.5 ppbv and typically requires VOC enrichment by concentrating up to one liter of a sample volume. The VOC concentration range for ambient air in many cases includes the concentration at which continuous exposure over a lifetime is estimated to constitute a 10<sup>-6</sup> or higher lifetime risk of developing cancer in humans. Under circumstances in which many hazardous VOCs are present at 10<sup>-6</sup> risk concentrations, the total risk may be significantly greater.
- 1.3 This method applies under most conditions encountered in sampling of ambient air into canisters. However, the composition of a gas mixture in a canister, under unique or unusual conditions, will change so that the sample is known not to be a true representation of the ambient air from which it was taken. For example, low humidity conditions in the sample may lead to losses of certain VOCs on the canister walls, losses that would not happen if the humidity were higher. If the canister is pressurized, then condensation of water from high humidity samples may cause fractional losses of water-soluble compounds. Since the canister surface area is limited, all gases are in competition for the available active sites. Hence an absolute storage stability cannot be assigned to a specific gas. Fortunately, under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations after storage times of up to thirty days (see Section 8).
- 1.4 Use of the Compendium Method TO-15 for many of the VOCs listed in Table 1 is likely to present two difficulties: (1) what calibration standard to use for establishing a basis for testing and quantitation, and (2) how

Method TO-15 VOCs

to obtain an audit standard. In certain cases a chemical similarity exists between a thoroughly tested compound and others on the Title III list. In this case, what works for one is likely to work for the other in terms of making standards. However, this is not always the case and some compound standards will be troublesome. The reader is referred to the Section 9.2 on standards for guidance. Calibration of compounds such as formaldehyde, diazomethane, and many of the others represents a challenge.

- 1.5 Compendium Method TO-15 should be considered for use when a subset of the 97 Title III VOCs constitute the target list. Typical situations involve ambient air testing associated with the permitting procedures for emission sources. In this case sampling and analysis of VOCs is performed to determine the impact of dispersing source emissions in the surrounding areas. Other important applications are prevalence and trend monitoring for hazardous VOCs in urban areas and risk assessments downwind of industrialized or source-impacted areas.
- 1.6 Solid adsorbents can be used in lieu of canisters for sampling of VOCs, provided the solid adsorbent packings, usually multisorbent packings in metal or glass tubes, can meet the performance criteria specified in Compendium Method TO-17 which specifically addresses the use of multisorbent packings. The two sample collection techniques are different but become the same upon movement of the sample from the collection medium (canister or multisorbent tubes) onto the sample concentrator. Sample collection directly from the atmosphere by automated gas chromatographs can be used in lieu of collection in canisters or on solid adsorbents.

#### 2. Summary of Method

- 2.1 The atmosphere is sampled by introduction of air into a specially-prepared stainless steel canister. Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. A pump ventilated sampling line is used during sample collection with most commercially available samplers. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister.
- 2.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to the laboratory for analysis.
- 2.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is stored until analysis. Storage times of up to thirty days have been demonstrated for many of the VOCs (5).
- 2.4 To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. A portion of the water vapor in the sample breaks through the concentrator during sampling, to a degree depending on the multisorbent composition, duration of sampling, and other factors. Water content of the sample can be further reduced by dry purging the concentrator with helium while retaining target compounds. After the concentration and drying steps are completed, the VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a reduced temperature trap or small volume multisorbent trap. The sample is then released by thermal desorption and carried onto a gas chromatographic column for separation.

As a simple alternative to the multisorbent/dry purge water management technique, the amount of water vapor in the sample can be reduced below any threshold for affecting the proper operation of the analytical system by

VOCs Method TO-15

reducing the sample size. For example, a small sample can be concentrated on a cold trap and released directly to the gas chromatographic column. The reduction in sample volume may require an enhancement of detector sensitivity.

Other water management approaches are also acceptable as long as their use does not compromise the attainment of the performance criteria listed in Section 11. A listing of some commercial water management systems is provided in Appendix A. One of the alternative ways to dry the sample is to separate VOCs from condensate on a low temperature trap by heating and purging the trap.

2.5 The analytical strategy for Compendium Method TO-15 involves using a high resolution gas chromatograph (GC) coupled to a mass spectrometer. If the mass spectrometer is a linear quadrupole system, it is operated either by continuously scanning a wide range of mass to charge ratios (SCAN mode) or by monitoring select ion monitoring mode (SIM) of compounds on the target list. If the mass spectrometer is based on a standard ion trap design, only a scanning mode is used (note however, that the Selected Ion Storage (SIS) mode for the ion trap has features of the SIM mode). Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This establishes the compound concentration that exists in the sample.

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification. If the technique is supported by a comprehensive mass spectral database and a knowledgeable operator, then the correct identification and quantification of VOCs is further enhanced.

#### 3. Significance

- **3.1** Compendium Method TO-15 is significant in that it extends the Compendium Method TO-14A description for using canister-based sampling and gas chromatographic analysis in the following ways:
  - Compendium Method TO-15 incorporates a multisorbent/dry purge technique or equivalent (see Appendix A) for water management thereby addressing a more extensive set of compounds (the VOCs mentioned in Title III of the CAAA of 1990) than addressed by Compendium Method TO-14A. Compendium Method TO-14A approach to water management alters the structure or reduces the sample stream concentration of some VOCs, especially water-soluble VOCs.
  - Compendium Method TO-15 uses the GC/MS technique as the only means to identify and quantitate target compounds. The GC/MS approach provides a more scientifically-defensible detection scheme which is generally more desirable than the use of single or even multiple specific detectors.
  - In addition, Compendium Method TO-15 establishes method performance criteria for acceptance of data, allowing the use of alternate but equivalent sampling and analytical equipment. There are several new and viable commercial approaches for water management as noted in Appendix A of this method on which to base a VOC monitoring technique as well as other approaches to sampling (i.e., autoGCs and solid

Method TO-15 VOCs

adsorbents) that are often used. This method lists performance criteria that these alternatives must meet to be acceptable alternatives for monitoring ambient VOCs.

- Finally, Compendium Method TO-15 includes enhanced provisions for inherent quality control. The method uses internal analytical standards and frequent verification of analytical system performance to assure control of the analytical system. This more formal and better documented approach to quality control guarantees a higher percentage of good data.
- 3.2 With these features, Compendium Method TO-15 is a more general yet better defined method for VOCs than Compendium Method TO-14A. As such, the method can be applied with a higher confidence to reduce the uncertainty in risk assessments in environments where the hazardous volatile gases listed in the Title III of the Clean Air Act Amendments of 1990 are being monitored. An emphasis on risk assessments for human health and effects on the ecology is a current goal for the U.S. EPA.

#### 4. Applicable Documents

#### 4.1 ASTM Standards

- Method D1356 Definitions of Terms Relating to Atmospheric Sampling and Analysis.
- Method E260 Recommended Practice for General Gas Chromatography Procedures.
- Method E355 Practice for Gas Chromatography Terms and Relationships.
- Method D5466 Standard Test Method of Determination of Volatile Organic Compounds in Atmospheres (Canister Sampling Methodology).

#### 4.2 EPA Documents

- Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, U. S. Environmental Protection Agency, EPA-600/R-94-038b, May 1994.
- Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air, U. S. Environmental Protection Agency, EPA-600/4-83-027, June 1983.
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-14, Second Supplement, U. S. Environmental Protection Agency, EPA-600/4-89-018, March 1989.
- Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites, U. S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C., Draft Report, June 1990.
- · Clean Air Act Amendments of 1990, U. S. Congress, Washington, D.C., November 1990.

## 5. Definitions

[Note: Definitions used in this document and any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. Aside from the definitions given below, all pertinent abbreviations and symbols are defined within this document at point of use.]

**5.1** Gauge Pressure—pressure measured with reference to the surrounding atmospheric pressure, usually expressed in units of kPa or psi. Zero gauge pressure is equal to atmospheric (barometric) pressure.

**5.2 Absolute Pressure**—pressure measured with reference to absolute zero pressure, usually expressed in units of kPa, or psi.

- 5.3 Cryogen—a refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Typical cryogens are liquid nitrogen (bp -195.8°C), liquid argon (bp -185.7°C), and liquid CO<sub>2</sub> (bp -79.5°C).
- **5.4 Dynamic Calibration**—calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system from a manifold through which the gas standards are flowing.
- 5.5 **Dynamic Dilution**—means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continuously blended with humidified zero air in a manifold so that a flowing stream of calibration mixture is available at the inlet of the analytical system.
- 5.6 MS-SCAN—mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.
- 5.7 MS-SIM—mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].
- **5.8 Qualitative Accuracy**—the degree of measurement accuracy required to correctly identify compounds with an analytical system.
- **5.9 Quantitative Accuracy**—the degree of measurement accuracy required to correctly measure the concentration of an identified compound with an analytical system with known uncertainty.
- **5.10 Replicate Precision**—precision determined from two canisters filled from the same air mass over the same time period and determined as the absolute value of the difference between the analyses of canisters divided by their average value and expressed as a percentage (see Section 11 for performance criteria for replicate precision).
- **5.11 Duplicate Precision**—precision determined from the analysis of two samples taken from the same canister. The duplicate precision is determined as the absolute value of the difference between the canister analyses divided by their average value and expressed as a percentage.
- 5.12 Audit Accuracy—the difference between the analysis of a sample provided in an audit canister and the nominal value as determined by the audit authority, divided by the audit value and expressed as a percentage (see Section 11 for performance criteria for audit accuracy).

#### 6. Interferences and Contamination

6.1 Very volatile compounds, such as chloromethane and vinyl chloride can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas. Refocusing of the sample after collection on the primary trap, either on a separate focusing trap or at the head of the gas chromatographic column, mitigates this problem.

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6.2 Interferences in canister samples may result from improper use or from contamination of: (1) the canisters due to poor manufacturing practices, (2) the canister cleaning apparatus, and (3) the sampling or analytical system. Attention to the following details will help to minimize the possibility of contamination of canisters.

- **6.2.1** Canisters should be manufactured using high quality welding and cleaning techniques, and new canisters should be filled with humidified zero air and then analyzed, after "aging" for 24 hours, to determine cleanliness. The cleaning apparatus, sampling system, and analytical system should be assembled of clean, high quality components and each system should be shown to be free of contamination.
- **6.2.2** Canisters should be stored in a contaminant-free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample.
- 6.2.3 Impurities in the calibration dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components must be avoided.
- **6.2.4** Significant contamination of the analytical equipment can occur whenever samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration (>25 ppbv of a trace species) sample is encountered, it should be followed by an analysis of humid zero air to check for carry-over contamination.
- 6.2.5 In cases when solid sorbents are used to concentrate the sample prior to analysis, the sorbents should be tested to identify artifact formation (see Compendium Method TO-17 for more information on artifacts).

#### 7. Apparatus and Reagents

[Note: Compendium Method To-14A list more specific requirements for sampling and analysis apparatus which may be of help in identifying options. The listings below are generic:]

#### 7.1 Sampling Apparatus

[Note: Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been used as part of U.S. Environmental Protection Agency's Toxic Air Monitoring Stations (TAMS), Urban Air Toxic Monitoring Program (UATMP), the non-methane organic compound (NMOC) sampling and analysis program, and the Photochemical Assessment Monitoring Stations (PAMS).]

- 7.1.1 Subatmospheric Pressure (see Figure 1, without metal bellows type pump).
  - 7.1.1.1 Sampling Inlet Line. Stainless steel tubing to connect the sampler to the sample inlet.
- 7.1.1.2 Sample Canister. Leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and specially prepared interior surfaces (see Appendix B for a listing of known manufacturers/resellers of canisters).
- 7.1.1.3 Stainless Steel Vacuum/Pressure Gauges. Two types are required, one capable of measuring vacuum (-100 to 0 kPa or 0 to 30 in Hg) and pressure (0-206 kPa or 0-30 psig) in the sampling system and a second type (for checking the vacuum of canisters during cleaning) capable of measuring at 0.05 mm Hg (see Appendix B) within 20%. Gauges should be tested clean and leak tight.
- 7.1.1.4 Electronic Mass Flow Controller. Capable of maintaining a constant flow rate ( $\pm$  10%) over a sampling period of up to 24 hours and under conditions of changing temperature (20–40°C) and humidity.
  - 7.1.1.5 Particulate Matter Filter. 2- $\mu$ m sintered stainless steel in-line filter.

- 7.1.1.6 Electronic Timer. For unattended sample collection.
- 7.1.1.7 Solenoid Valve. Electrically-operated, bi-stable solenoid valve with Viton® seat and O-rings. A Skinner Magnelatch valve is used for purposes of illustration in the text (see Figure 2).
- 7.1.1.8 Chromatographic Grade Stainless Steel Tubing and Fittings. For interconnections. All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel or equivalent.
- **7.1.1.9** Thermostatically Controlled Heater. To maintain above ambient temperature inside insulated sampler enclosure.
  - 7.1.1.10 Heater Thermostat. Automatically regulates heater temperature.
  - 7.1.1.11 Fan. For cooling sampling system.
  - 7.1.1.12 Fan Thermostat. Automatically regulates fan operation.
- **7.1.1.13 Maximum-Minimum Thermometer**. Records highest and lowest temperatures during sampling period.
  - 7.1.1.14 Stainless Steel Shut-off Valve. Leak free, for vacuum/pressure gauge.
- 7.1.1.15 Auxiliary Vacuum Pump. Continuously draws air through the inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted.

[Note: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

- 7.1.1.16 Elapsed Time Meter. Measures duration of sampling.
- 7.1.1.17 Optional Fixed Orifice, Capillary, or Adjustable Micrometering Valve. May be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.
  - 7.1.2 Pressurized (see Figure 1 with metal bellows type pump and Figure 3).
- 7.1.2.1 Sample Pump. Stainless steel, metal bellows type, capable of 2 atmospheres output pressure. Pump must be free of leaks, clean, and uncontaminated by oil or organic compounds.

[Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Institute of Science and Technology, 20000 N.W. Walker Rd., Beaverton, Oregon 97006, 503-690-1077, and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensation flow restrictive device. In this configuration the pump is purged with a large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet.]

7.1.2.2 Other Supporting Materials. All other components of the pressurized sampling system are similar to components discussed in Sections 7.1.1.1 through 7.1.1.17.

#### 7.2 Analytical Apparatus

- 7.2.1 Sampling/Concentrator System (many commercial alternatives are available).
- **7.2.1.1 Electronic Mass Flow Controllers**. Used to maintain constant flow (for purge gas, carrier gas and sample gas) and to provide an analog output to monitor flow anomalies.
- **7.2.1.2 Vacuum Pump**. General purpose laboratory pump, capable of reducing the downstream pressure of the flow controller to provide the pressure differential necessary to maintain controlled flow rates of sample air.
- 7.2.1.3 Stainless Steel Tubing and Stainless Steel Fittings. Coated with fused silica to minimize active adsorption sites.

- **7.2.1.4 Stainless Steel Cylinder Pressure Regulators**. Standard, two-stage cylinder regulators with pressure gauges.
  - 7.2.1.5 Gas Purifiers. Used to remove organic impurities and moisture from gas streams.
  - 7.2.1.6 Six-port Gas Chromatographic Valve. For routing sample and carrier gas flows.
- **7.2.1.7 Multisorbent Concentrator**. Solid adsorbent packing with various retentive properties for adsorbing trace gases are commercially available from several sources. The packing contains more than one type of adsorbent packed in series.
- 7.2.1.7.1A pre-packed adsorbent trap (Supelco 2-0321) containing 200 mg Carbopack B (60/80 mesh) and 50 mg Carbosieve S-III (60/80 mesh) has been found to retain VOCs and allow some water vapor to pass through (6). The addition of a dry purging step allows for further water removal from the adsorbent trap. The steps constituting the dry purge technique that are normally used with multisorbent traps are illustrated in Figure 4. The optimum trapping and dry purging procedure for the Supelco trap consists of a sample volume of 320 mL and a dry nitrogen purge of 1300 mL. Sample trapping and drying is carried out at 25°C. The trap is back-flushed with helium and heated to 220°C to transfer material onto the GC column. A trap bake-out at 260°C for 5 minutes is conducted after each run.
- 7.2.1.7.2An example of the effectiveness of dry purging is shown in Figure 5. The multisorbent used in this case is Tenax/Ambersorb 340/Charcoal (7). Approximately 20% of the initial water content in the sample remains after sampling 500 mL of air. The detector response to water vapor (hydrogen atoms detected by atomic emission detection) is plotted versus purge gas volume. Additional water reduction by a factor of 8 is indicated at temperatures of 45°C or higher. Still further water reduction is possible using a two-stage concentration/dryer system.
- 7.2.1.8 Cryogenic Concentrator. Complete units are commercially available from several vendor sources. The characteristics of the latest concentrators include a rapid, "ballistic" heating of the concentrator to release any trapped VOCs into a small carrier gas volume. This facilitates the separation of compounds on the gas chromatographic column.
  - 7.2.2 Gas Chromatographic/Mass Spectrometric (GC/MS) System.
- 7.2.2.1 Gas Chromatograph. The gas chromatographic (GC) system must be capable of temperature programming. The column oven can be cooled to subambient temperature (e.g., -50°C) at the start of the gas chromatographic run to effect a resolution of the very volatile organic compounds. In other designs, the rate of release of compounds from the focusing trap in a two stage system obviates the need for retrapping of compounds on the column. The system must include or be interfaced to a concentrator and have all required accessories including analytical columns and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with Buna-N rubber components must not be used.
- 7.2.2.2 Chromatographic Columns. 100% methyl silicone or 5% phenyl, 95% methyl silicone fused silica capillary columns of 0.25- to 0.53-mm I.D. of varying lengths are recommended for separation of many of the possible subsets of target compounds involving nonpolar compounds. However, considering the diversity of the target list, the choice is left to the operator subject to the performance standards given in Section 11.
- 7.2.2.3 Mass Spectrometer. Either a linear quadrupole or ion trap mass spectrometer can be used as long as it is capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng or less of p-bromofluorobenzene (BFB) is analyzed.
- 7.2.2.3.1Linear Quadrupole Technology. A simplified diagram of the heart of the quadrupole mass spectrometer is shown in Figure 6. The quadrupole consists of a parallel set of four rod electrodes mounted in a square configuration. The field within the analyzer is created by coupling opposite pairs of rods together and applying radiofrequency (RF) and direct current (DC) potentials between the pairs of rods. Ions created in the ion source from the reaction of column eluates with electrons from the electron source are moved through the

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parallel array of rods under the influence of the generated field. Ions which are successfully transmitted through the quadrupole are said to possess stable trajectories and are subsequently recorded with the detection system. When the DC potential is zero, a wide band of m/z values is transmitted through the quadrupole. This "RF only" mode is referred to as the "total-ion" mode. In this mode, the quadrupole acts as a strong focusing lens analogous to a high pass filter. The amplitude of the RF determines the low mass cutoff. A mass spectrum is generated by scanning the DC and RF voltages using a fixed DC/RF ratio and a constant drive frequency or by scanning the frequency and holding the DC and RF constant. With the quadrupole system only 0.1 to 0.2 percent of the ions formed in the ion source actually reach the detector.

7.2.2.3.2Ion Trap Technology. An ion-trap mass spectrometer consists of a chamber formed between two metal surfaces in the shape of a hyperboloid of one sheet (ring electrode) and a hyperboloid of two sheets (the two end-cap electrodes). Ions are created within the chamber by electron impact from an electron beam admitted through a small aperture in one of the end caps. Radio frequency (RF) (and sometimes direct current voltage offsets) are applied between the ring electrode and the two end-cap electrodes establishing a quadrupole electric field. This field is uncoupled in three directions so that ion motion can be considered independently in each direction; the force acting upon an ion increases with the displacement of the ion from the center of the field but the direction of the force depends on the instantaneous voltage applied to the ring electrode. A restoring force along one coordinate (such as the distance, r, from the ion-trap's axis of radial symmetry) will exist concurrently with a repelling force along another coordinate (such as the distance, z, along the ion traps axis), and if the field were static the ions would eventually strike an electrode. However, in an RF field the force along each coordinate alternates direction so that a stable trajectory may be possible in which the ions do not strike a surface. In practice, ions of appropriate mass-to-charge ratios may be trapped within the device for periods of milliseconds to hours. A diagram of a typical ion trap is illustrated in Figure 7. Analysis of stored ions is performed by increasing the RF voltage, which makes the ions successively unstable. The effect of the RF voltage on the ring electrode is to "squeeze" the ions in the xy plane so that they move along the z axis. Half the ions are lost to the top cap (held at ground potential); the remaining ions exit the lower end cap to be detected by the electron multiplier. As the energy applied to the ring electrode is increased, the ions are collected in order of increasing mass to produce a conventional mass spectrum. With the ion trap, approximately 50 percent of the generated ions are detected. As a result, a significant increase in sensitivity can be achieved when compared to a full scan linear quadrupole system.

7.2.2.4 GC/MS Interface. Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points for each of the analytes of interest and can be used to achieve all acceptable performance criteria may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass, glass-lined, or fused silica-lined materials are recommended. Glass and fused silica should be deactivated.

7.2.2.5 Data System. The computer system that is interfaced to the mass spectrometer must allow the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as a Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, software must be available that allows for the comparison of sample spectra with reference library spectra. The National Institute of Standards and Technology (NIST) or Wiley Libraries or equivalent are recommended as reference libraries.

**7.2.2.6** Off-line Data Storage Device. Device must be capable of rapid recording and retrieval of data and must be suitable for long-term, off-line data storage.

#### 7.3 Calibration System and Manifold Apparatus (see Figure 8)

- 7.3.1 Calibration Manifold. Stainless steel, glass, or high purity quartz manifold, (e.g.,1.25-cm I.D. x 66-cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing. The manifold should be heated to ~50°C.
  - 7.3.2 Humidifier. 500-mL impinger flask containing HPLC grade deionized water.
- 7.3.3 Electronic Mass Flow Controllers. One 0 to 5 L/min unit and one or more 0 to 100 mL/min units for air, depending on number of cylinders in use for calibration.
  - 7.3.4 Teflon Filter(s). 47-mm Teflon® filter for particulate collection.

#### 7.4 Reagents

- 7.4.1 Neat Materials or Manufacturer-Certified Solutions/Mixtures. Best source (see Section 9).
- 7.4.2 Helium and Air. Ultra-high purity grade in gas cylinders. He is used as carrier gas in the GC.
- 7.4.3 Liquid Nitrogen or Liquid Carbon Dioxide. Used to cool secondary trap.
- 7.4.4 Deionized Water. High performance liquid chromatography (HPLC) grade, ultra-high purity (for humidifier).

#### 8. Collection of Samples in Canisters

#### 8.1 Introduction

- **8.1.1** Canister samplers, sampling procedures, and canister cleaning procedures have not changed very much from the description given in the original Compendium Method TO-14. Much of the material in this section is therefore simply a restatement of the material given in Compendium Method TO-14, repeated here in order to have all the relevant information in one place.
- 8.1.2 Recent notable additions to the canister technology has been in the application of canister-based systems for example, to microenvironmental monitoring (8), the capture of breath samples (9), and sector sampling to identify emission sources of VOCs (10).
- 8.1.3 EPA has also sponsored the development of a mathematical model to predict the storage stability of arbitrary mixtures of trace gases in humidified air (3), and the investigation of the SilcoSteel<sup>TM</sup> process of coating the canister interior with a film of fused silica to reduce surface activity (11). A recent summary of storage stability data for VOCs in canisters is given in the open literature (5).

#### 8.2 Sampling System Description

### 8.2.1 Subatmospheric Pressure Sampling [see Figure 1 (without metal bellows type pump)].

- **8.2.1.1** In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg (see Appendix C for discussion of evacuation pressure). When the canister is opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-weighted-average (TWA) samples (duration of 1-24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).
- **8.2.1.2** With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

# 8.2.2 Pressurized Sampling [see Figure 1 (with metal bellows type pump)].

- **8.2.2.1** Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 101-202 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canister can be filled at 10 mL/min for 24 hours to achieve a final pressure of 144 kPa (21 psig).
- **8.2.2.2** In pressurized canister sampling, a metal bellows type pump draws in air from the sampling manifold to fill and pressurize the sample canister.

#### 8.2.3 All Samplers.

**8.2.3.1** A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled (to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling) over the desired sample period. The flow rate can be calculated by:

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate, mL/min.

P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa gauge}}{101.2}$$
 +

V = volume of the canister, mL.

T =sample period, hours.

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ mL/min}$$

- **8.2.3.2** For automatic operation, the timer is designed to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and to close the valve when stopping the pump.
- 8.2.3.3 The use of the Skinner Magnelatch valve (see Figure 2) avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton® valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with a conventional mechanical timer and a special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid valve with a conventional mechanical timer is illustrated in Figure 2(a). However, with this simple circuit, the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better circuit incorporating a time-delay relay to provide more reliable valve operation is shown in Figure 2(b).

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- **8.2.3.4** The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period.
- **8.2.3.5** As an option, a second electronic timer may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.
- **8.2.3.6** Prior to field use, each sampling system must pass a humid zero air certification (see Section 8.4.3). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 8.4.1).

#### 8.3 Sampling Procedure

- **8.3.1** The sample canister should be cleaned and tested according to the procedure in Section 8.4.1.
- **8.3.2** A sample collection system is assembled as shown in Figures 1 and 3 and must be cleaned according to the procedure outlined in Sections 8.4.2 and 8.4.4.

[Note: The sampling system should be contained in an appropriate enclosure.]

- **8.3.3** Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B of Compendium Method TO-14A, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable GC screening analysis would be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.
- **8.3.4** After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on the canister sampling field test data sheet (FTDS), as documented in Figure 9.

[Note: The following discussion is related to Figure 1]

8.3.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system.

[Note: For a subatmospheric sampler, a flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.]

A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within  $\pm 10\%$ . If not, the sampler mass flow meter needs to be recalibrated or there is a leak in the system. This should be investigated and corrected.

[Note: Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate to compensate for any zero drift.]

After 2 minutes, the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 3.5 mL/min for 24 hr, 7.0 mL/min for 12 hr). Record final flow under "CANISTER FLOW RATE" on the FTDS.

**8.3.6** The sampler is turned off and the elapsed time meter is reset to 000.0.

[Note: Whenever the sampler is turned off, wait at least 30 seconds to turn the sampler back on.]

- **8.3.7** The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 8.4.1) canister is attached to the system.
  - **8.3.8** The canister valve and vacuum/pressure gauge valve are opened.
- **8.3.9** Pressure/vacuum in the canister is recorded on the canister FTDS (see Figure 9) as indicated by the sampler vacuum/pressure gauge.
- **8.3.10** The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer is reset to current temperature. Time of day and elapsed time meter readings are recorded on the canister FTDS.
- **8.3.11** The electronic timer is set to start and stop the sampling period at the appropriate times. Sampling starts and stops by the programmed electronic timer.
- **8.3.12** After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the FTDS. The current reading from the flow controller is recorded.
- **8.3.13** At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the FTDS. Pressure should be close to desired pressure.

[Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.]

Time of day and elapsed time meter readings are also recorded.

**8.3.14** The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once again connected to the inlet manifold in front of the in-line filter and a "practice" canister is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister FTDS (see Figure 9).

[Note: For a pressurized system, the final flow may be measured directly.]

The sampler is turned off.

**8.3.15** An identification tag is attached to the canister. Canister serial number, sample number, location, and date, as a minimum, are recorded on the tag. The canister is routinely transported back to the analytical laboratory with other canisters in a canister shipping case.

#### 8.4 Cleaning and Certification Program

- 8.4.1 Canister Cleaning and Certification.
  - **8.4.1.1** All canisters must be clean and free of any contaminants before sample collection.
- **8.4.1.2** All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air.

[Note: The canister cleaning system in Figure 10 can be used for this task.]

The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If acceptable, the pressure should not vary more than  $\pm$  13.8 kPa ( $\pm$  2 psig) over the 24 hour period.

**8.4.1.3** A canister cleaning system may be assembled as illustrated in Figure 10. Cryogen is added to both the vacuum pump and zero air supply traps. The canister(s) are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The vacuum pump is started and the vent shut-off valve is then closed and the vacuum shut-off valve is opened. The canister(s) are evacuated to <0.05 mm Hg (see Appendix B) for at least 1 hour.

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[Note: On a daily basis or more often if necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.]

Air released/evacuated from canisters should be diverted to a fume hood.

- **8.4.1.4** The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.
- **8.4.1.5** The zero air shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent shut-off valve is closed. Repeat Sections 8.4.1.3 through 8.4.1.5 two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.
- **8.4.1.6** At the end of the evacuation/pressurization cycle, the canister is pressurized to 206 kPa (30 psig) with humid zero air. The canister is then analyzed by a GC/MS analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used. As a "blank" check of the canister(s) and cleanup procedure, the final humid zero air fill of 100% of the canisters is analyzed until the cleanup system and canisters are proven reliable (less than 0.2 ppbv of any target VOCs). The check can then be reduced to a lower percentage of canisters.
- **8.4.1.7** The canister is reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg (see Appendix B) and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting. The canister is now ready for collection of an air sample. An identification tag is attached to the inlet of each canister for field notes and chain-of-custody purposes. An alternative to evacuating the canister at this point is to store the canisters and reevacuate them just prior to the next use.
- **8.4.1.8** As an option to the humid zero air cleaning procedures, the canisters are heated in an isothermal oven not to exceed 100°C during evacuation of the canister to ensure that higher molecular weight compounds are not retained on the walls of the canister.

[Note: For sampling more complex VOC mixtures the canisters should be heated to higher temperatures during the cleaning procedure although a special high temperature valve would be needed].

Once heated, the canisters are evacuated to <0.05 mm Hg (see Appendix B) and maintained there for 1 hour. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by a GC/MS system after a minimum of 12 hrs of "aging." Any canister that has not tested clean (less than 0.2 ppbv each of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to <0.05 mm Hg (see Appendix B) and remain in the evacuated state until used. As noted in Section 8.4.1.7, reevacuation can occur just prior to the next use.

## 8.4.2 Cleaning Sampling System Components.

- **8.4.2.1** Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.
- **8.4.2.2** The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.
  - **8.4.2.3** Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.
  - 8.4.3 Zero Air Certification.

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[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv each of target compounds) have occurred when challenged with the test gas stream.]

- **8.4.3.1** The cleanliness of the sampling system is determined by testing the sampler with humid zero air without an evacuated gas sampling canister, as follows.
- **8.4.3.2** The calibration system and manifold are assembled, as illustrated in Figure 8. The sampler (without an evacuated gas canister) is connected to the manifold and the zero air cylinder is activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].
- **8.4.3.3** The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to the water management system/VOC preconcentrator of an analytical system.

[Note: The exit of the sampling system (without the canister) replaces the canister in Figure 11.]

After the sample volume (e.g., 500 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed and refocussed on a cold trap. This trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. The VOCs are refocussed prior to gas chromatographic separation. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC/MS (see Section 10) system. The analytical system should not detect greater than 0.2 ppbv of any targeted VOCs in order for the sampling system to pass the humid zero air certification test. Chromatograms (using an FID) of a certified sampler and contaminated sampler are illustrated in Figures 12(a) and 12(b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in Section 8.4.4.

- 8.4.4 Sampler System Certification with Humid Calibration Gas Standards from a Dynamic Calibration System
  - **8.4.4.1** Assemble the dynamic calibration system and manifold as illustrated in Figure 8.
- **8.4.4.2** Verify that the calibration system is clean (less than 0.2 ppbv of any target compounds) by sampling a humidified gas stream, <u>without</u> gas calibration standards, with a previously certified clean canister (see Section 8.1).
- **8.4.4.3** The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of any targeted compounds is found.
- **8.4.4.4** For generating the humidified calibration standards, the calibration gas cylinder(s) containing nominal concentrations of 10 ppmv in nitrogen of selected VOCs is attached to the calibration system as illustrated in Figure 8. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 mL/min certified mass flow controllers to generate ppb levels of calibration standards.
- **8.4.4.5** After the appropriate equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(b).
  - **8.4.4.6** Sample the dynamic calibration gas stream with the sampling system.
- **8.4.4.7** Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC/MS analytical system [Figure 8(a)] to provide reference concentrations of generated VOCs.
- **8.4.4.8** At the end of the sampling period (normally the same time period used for experiments), the sampling system canister is analyzed and compared to the reference GC/MS analytical system to determine if the concentration of the targeted VOCs was increased or decreased by the sampling system.
  - 8.4.4.9 A recovery of between 90% and 110% is expected for all targeted VOCs.
  - 8.4.5 Sampler System Certification without Compressed Gas Cylinder Standards.

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**8.4.5.1** Not all the gases on the Title III list are available/compatible with compressed gas standards. In these cases sampler certification must be approached by different means.

**8.4.5.2** Definitive guidance is not currently available in these cases; however, Section 9.2 lists several ways to generate gas standards. In general, Compendium Method TO-14A compounds (see Table 1) are available commercially as compressed gas standards.

#### 9. GC/MS Analysis of Volatiles from Canisters

#### 9.1 Introduction

- 9.1.1 The analysis of canister samples is accomplished with a GC/MS system. Fused silica capillary columns are used to achieve high temporal resolution of target compounds. Linear quadrupole or ion trap mass spectrometers are employed for compound detection. The heart of the system is composed of the sample inlet concentrating device that is needed to increase sample loading into a detectable range. Two examples of concentrating systems are discussed. Other approaches are acceptable as long as they are compatible with achieving the system performance criteria given in Section 11.
- 9.1.2 With the first technique, a whole air sample from the canister is passed through a multisorbent packing (including single adsorbent packings) contained within a metal or glass tube maintained at or above the surrounding air temperature. Depending on the water retention properties of the packing, some or most of the water vapor passes completely through the trap during sampling. Additional drying of the sample is accomplished after the sample concentration is completed by forward purging the trap with clean, dry helium or another inert gas (air is not used). The sample is then thermally desorbed from the packing and backflushed from the trap onto a gas chromatographic column. In some systems a "refocusing" trap is placed between the primary trap and the gas chromatographic column. The specific system design downstream of the primary trap depends on technical factors such as the rate of thermal desorption and sampled volume, but the objective in most cases is to enhance chromatographic resolution of the individual sample components before detection on a mass spectrometer.
- 9.1.3 Sample drying strategies depend on the target list of compounds. For some target compound lists, the multisorbent packing of the concentrator can be selected from hydrophobic adsorbents which allow a high percentage of water vapor in the sample to pass through the concentrator during sampling and without significant loss of the target compounds. However, if very volatile organic compounds are on the target list, the adsorbents required for their retention may also strongly retain water vapor and a more lengthy dry purge is necessary prior to analysis.
- 9.1.4 With the second technique, a whole air sample is passed through a concentrator where the VOCs are condensed on a reduced temperature surface (cold trap). Subsequently, the condensed gases are thermally desorbed and backflushed from the trap with an inert gas onto a gas chromatographic column. This concentration technique is similar to that discussed in Compendium Method TO-14, although a membrane dryer is not used. The sample size is reduced in volume to limit the amount of water vapor that is also collected (100 mL or less may be necessary). The attendant reduction in sensitivity is offset by enhancing the sensitivity of detection, for example by using an ion trap detector.

#### 9.2 Preparation of Standards

#### 9.2.1 Introduction.

- 9.2.1.1 When available, standard mixtures of target gases in high pressure cylinders must be certified traceable to a NIST Standard Reference Material (SRM) or to a NIST/EPA approved Certified Reference Material (CRM). Manufacturer's certificates of analysis must be retained to track the expiration date.
- **9.2.1.2** The neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.
- **9.2.1.3** Cylinder(s) containing approximately 10 ppmv of each of the target compounds are typically used as primary stock standards. The components may be purchased in one cylinder or in separate cylinders depending on compatibility of the compounds and the pressure of the mixture in the cylinder. Refer to manufacturer's specifications for guidance on purchasing and mixing VOCs in gas cylinders.

## 9.2.2 Preparing Working Standards.

- **9.2.2.1** Instrument Performance Check Standard. Prepare a standard solution of BFB in humidified zero air at a concentration which will allow collection of 50 ng of BFB or less under the optimized concentration parameters.
- **9.2.2.2 Calibration Standards**. Prepare five working calibration standards in humidified zero air at a concentration which will allow collection at the 2, 5, 10, 20, and 50 ppbv level for each component under the optimized concentration parameters.
- 9.2.2.3 Internal Standard Spiking Mixture. Prepare an internal spiking mixture containing bromochloromethane, chlorobenzene-d<sub>5</sub>, and 1,4-difluorobenzene at 10 ppmv each in humidified zero air to be added to the sample or calibration standard. 500 µL of this mixture spiked into 500 mL of sample will result in a concentration of 10 ppbv. The internal standard is introduced into the trap during the collection time for all calibration, blank, and sample analyses using the apparatus shown in Figure 13 or by equivalent means. The volume of internal standard spiking mixture added for each analysis must be the same from run to run.

#### 9.2.3 Standard Preparation by Dynamic Dilution Technique.

- 9.2.3.1 Standards may be prepared by dynamic dilution of the gaseous contents of a cylinder(s) containing the gas calibration stock standards with humidified zero air using mass flow controllers and a calibration manifold. The working standard may be delivered from the manifold to a clean, evacuated canister using a pump and mass flow controller.
- 9.2.3.2 Alternatively, the analytical system may be calibrated by sampling directly from the manifold if the flow rates are optimized to provide the desired amount of calibration standards. However, the use of the canister as a reservoir prior to introduction into the concentration system resembles the procedure normally used to collect samples and is preferred. Flow rates of the dilution air and cylinder standards (all expressed in the same units) are measured using a bubble meter or calibrated electronic flow measuring device, and the concentrations of target compounds in the manifold are then calculated using the dilution ratio and the original concentration of each compound.

Manifold Conc. = 
$$\frac{\text{(Original Conc.) (Std. Gas Flowrate)}}{\text{(Air Flowrate)} + \text{(Std. Gas Flowrate)}}$$

9.2.3.3 Consider the example of 1 mL/min flow of 10 ppmv standard diluted with 1,000 mL/min of humid air provides a nominal 10 ppbv mixture, as calculated below:

Manifold Conc. = 
$$\frac{(10 \text{ ppm})(1 \text{ mL/min})(1000 \text{ ppb/1 ppm})}{(1000 \text{ mL/min}) + (1 \text{ mL/min})} = 10 \text{ ppb}$$

### 9.2.4 Standard Preparation by Static Dilution Bottle Technique

[Note: Standards may be prepared in canisters by spiking the canister with a mixture of components prepared in a static dilution bottle (12). This technique is used specifically for liquid standards.]

- 9.2.4.1 The volume of a clean 2-liter round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density for the water of 1 g/mL, the weight of the water in grams is taken as the volume of the flask in milliliters.
- 9.2.4.2 The flask is flushed with helium by attaching a tubing into the glass neck to deliver the helium. After a few minutes, the tubing is removed and the glass neck is immediately closed with a Mininert septum cap.
- 9.2.4.3 The flask is placed in a 60°C oven and allowed to equilibrate at that temperature for about 15 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 60°C.
- 9.2.4.4 The contents are allowed to equilibrate in the oven for at least 30 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots to avoid condensation.
- 9.2.4.5 Sample aliquots may then be taken for introduction into the analytical system or for further dilution. An aliquot or aliquots totaling greater than 1 percent of the flask volume should be avoided.
- 9.2.4.6 Standards prepared by this method are stable for one week. The septum must be replaced with each freshly prepared standard.
  - 9.2.4.7 The concentration of each component in the flask is calculated using the following equation:

Concentration, mg/L = 
$$\frac{(V_a)(d)}{V_f}$$

where:

V<sub>a</sub> = Volume of liquid neat standard injected into the flask, μL.

d = Density of the liquid neat standard, mg/μL.

V<sub>f</sub> = Volume of the flask, L.

9.2.4.8 To obtain concentrations in ppbv, the equation given in Section 9.2.5.7 can be used.

[Note: In the preparation of standards by this technique, the analyst should make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask. Precautions should also be taken to avoid a significant decrease in pressure inside the flask after withdrawal of aliquot(s).]

#### 9.2.5 Standard Preparation Procedure in High Pressure Cylinders

[Note: Standards may be prepared in high pressure cylinders (13). A modified summary of the procedure is provided below.]

9.2.5.1 The standard compounds are obtained as gases or neat liquids (greater than 98 percent purity).

- 9.2.5.2 An aluminum cylinder is flushed with high-purity nitrogen gas and then evacuated to better than 25 in. Hg.
- 9.2.5.3 Predetermined amounts of each neat standard compound are measured using a microliter or gastight syringe and injected into the cylinder. The cylinder is equipped with a heated injection port and nitrogen flow to facilitate sample transfer.
  - 9.2.5.4 The cylinder is pressurized to 1000 psig with zero nitrogen.

[Note: User should read all SOPs associated with generating standards in high pressure cylinders. Follow all safety requirements to minimize danger from high pressure cylinders.]

- 9.2.5.5 The contents of the cylinder are allowed to equilibrate (~24 hrs) prior to withdrawal of aliquots into the GC system.
  - 9.2.5.6 If the neat standard is a gas, the cylinder concentration is determined using the following equation:

Concentration, ppbv = 
$$\frac{\text{Volume}_{\text{standard}}}{\text{Volume}_{\text{dilution gas}}} \times 10^9$$

[Note: Both values must be expressed in the same units.]

**9.2.5.7** If the neat standard is a liquid, the gaseous concentration can be determined using the following equations:

$$V = \frac{nRT}{P}$$

and:

$$n = \frac{(mL)(d)}{MW}$$

where:

V = Gaseous volume of injected compound at EPA standard temperature (25°C) and pressure (760 mm Hg), L.

n = Moles.

R = Gas constant, 0.08206 L-atm/mole °K.

T = 298°K (standard temperature).

P = 1 standard pressure, 760 mm Hg (1 atm).

mL = Volume of liquid injected, mL.

d = Density of the neat standard, g/mL.

MW = Molecular weight of the neat standard expressed, g/g-mole.

The gaseous volume of the injected compound is divided by the cylinder volume at STP and then multiplied by  $10^9$  to obtain the component concentration in ppb units.

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#### 9.2.6 Standard Preparation by Water Methods.

[Note: Standards may be prepared by a water purge and trap method (14) and summarized as follows].

9.2.6.1 A previously cleaned and evacuated canister is pressurized to 760 mm Hg absolute (1 atm) with zero grade air.

9.2.6.2 The air gauge is removed from the canister and the sparging vessel is connected to the canister with the short length of 1/16 in. stainless steel tubing.

[Note: Extra effort should be made to minimize possible areas of dead volume to maximize transfer of analytes from the water to the canister.]

- **9.2.6.3** A measured amount of the stock standard solution and the internal standard solution is spiked into 5 mL of water.
- 9.2.6.4 This water is transferred into the sparge vessel and purged with nitrogen for 10 mins at 100 mL/min. The sparging vessel is maintained at  $40^{\circ}\text{C}$ .
- 9.2.6.5 At the end of 10 mins, the sparge vessel is removed and the air gauge is re-installed, to further pressurize the canister with pure nitrogen to 1500 mm Hg absolute pressure (approximately 29 psia).
  - 9.2.6.6 The canister is allowed to equilibrate overnight before use.
  - 9.2.6.7 A schematic of this approach is shown in Figure 14.

#### 9.2.7 Preparation of Standards by Permeation Tubes.

- 9.2.7.1 Permeation tubes can be used to provide standard concentration of a trace gas or gases. The permeation of the gas can occur from inside a permeation tube containing the trace species of interest to an air stream outside. Permeation can also occur from outside a permeable membrane tube to an air stream passing through the tube (e.g., a tube of permeable material immersed in a liquid).
- 9.2.7.2 The permeation system is usually held at a constant temperature to generate a constant concentration of trace gas. Commercial suppliers provide systems for generation and dilution of over 250 compounds. Some commercial suppliers of permeation tube equipment are listed in Appendix D.

#### 9.2.8 Storage of Standards.

- **9.2.8.1** Working standards prepared in canisters may be stored for thirty days in an atmosphere free of potential contaminants.
  - 9.2.8.2 It is imperative that a storage logbook be kept to document storage time.

#### 10. GC/MS Operating Conditions

#### 10.1 Preconcentrator

The following are typical cryogenic and adsorbent preconcentrator analytical conditions which, however, depend on the specific combination of solid sorbent and must be selected carefully by the operator. The reader is referred to Tables 1 and 2 of Compendium Method TO-17 for guidance on selection of sorbents. An example of a system using a solid adsorbent preconcentrator with a cryofocusing trap is discussed in the literature (15). Oven temperature programming starts above ambient.

#### 10.1.1 Sample Collection Conditions

Cryogenic Trap

Adsorbent Trap

Set point -150°C Set point 27°C
Sample volume - up to 100 mL Sample volume - up to 1,000 mL
Carrier gas purge flow - none Carrier gas purge flow - selectable

[Note: The analyst should optimize the flow rate, duration of sampling, and absolute sample volume to be used. Other preconcentration systems may be used provided performance standards (see Section 11) are realized.]

#### 10.1.2 Desorption Conditions

Cryogenic Trap		Adsorbent Trap	
Desorb Temperature	120°C	Desorb Temperature	Variable
Desorb Flow Rate	~ 3 mL/min He	Desorb Flow Rate	~3 mL/min He
Desorb Time	<60 sec	Desorb Time	<60 sec

The adsorbent trap conditions depend on the specific solid adsorbents chosen (see manufacturers' specifications).

#### 10.1.3 Trap Reconditioning Conditions.

Cryogenic Trap		Adsorbent Trap	
Initial bakeout Variable (24 hrs)	120°C (24 hrs)	Initial bakeout	
After each run	120°C (5 min)	After each run	Variable (5 min)

### 10.2 GC/MS System

- 10.2.1 Optimize GC conditions for compound separation and sensitivity. Baseline separation of benzene and carbon tetrachloride on a 100% methyl polysiloxane stationary phase is an indication of acceptable chromatographic performance.
- 10.2.2 The following are the recommended gas chromatographic analytical conditions when using a 50-meter by 0.3-mm I.D., 1  $\mu$ m film thickness fused silica column with refocusing on the column.

<u>Item</u>	Condition	
Carrier Gas: Flow Rate: Temperature Program:	Helium Generally 1-3 mL/min as Initial Temperature: Initial Hold Time: Ramp Rate: Final Temperature: Final Hold Time:	s recommended by manufacturer -50°C 2 min 8° C/min 200°C Until all target compounds elute.

10.2.3 The following are the recommended mass spectrometer conditions:

Item	Condition	

Electron Energy: 70 Volts (nominal)

Mass Range: 35-300 amu [the choice of 35 amu excludes the detection of some target compounds

such as methanol and formaldehyde, and the quantitation of others such as ethylene oxide, ethyl carbamate, etc. (see Table 2). Lowering the mass range and using special programming features available on modern gas chromatographs will be necessary in

these cases, but are not considered here.

Scan Time: To give at least 10 scans per peak, not to exceed 1 second per scan].

A schematic for a typical GC/MS analytical system is illustrated in Figure 15.

## 10.3 Analytical Sequence

10.3.1 Introduction. The recommended GC/MS analytical sequence for samples during each 24-hour time period is as follows:

- Perform instrument performance check using bromofluorobenzene (BFB).
- Initiate multi-point calibration or daily calibration checks.
- · Perform a laboratory method blank.
- Complete this sequence for analysis of ≤20 field samples.

### 10.4 Instrument Performance Check

- 10.4.1 Summary. It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. The GC/MS system is set up according to the manufacturer's specifications, and the mass calibration and resolution of the GC/MS system are then verified by the analysis of the instrument performance check standard, bromofluorobenzene (BFB).
- 10.4.2 Frequency. Prior to the analyses of any samples, blanks, or calibration standards, the Laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard containing BFB. The instrument performance check solution must be analyzed initially and once per 24-hour time period of operation.

The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or daily calibration check criteria) begins at the injection of the BFB which the laboratory records as documentation of a compliance tune.

10.4.3 Procedure. The analysis of the instrument performance check standard is performed by trapping 50 ng of BFB under the optimized preconcentration parameters. The BFB is introduced from a cylinder into the GC/MS via a sample loop valve injection system similar to that shown in Figure 13.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.

- 10.4.4 Technical Acceptance Criteria. Prior to the analysis of any samples, blanks, or calibration standards, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard as specified in Table 3.
- 10.4.5 Corrective Action. If the BFB acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other necessary actions to achieve the acceptance criteria.

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10.4.6 Documentation. Results of the BFB tuning are to be recorded and maintained as part of the instrumentation log.

#### 10.5 Initial Calibration

10.5.1 Summary. Prior to the analysis of samples and blanks but after the instrument performance check standard criteria have been met, each GC/MS system must be calibrated at five concentrations that span the monitoring range of interest in an initial calibration sequence to determine instrument sensitivity and the linearity of GC/MS response for the target compounds. For example, the range of interest may be 2 to 20 ppbv, in which case the five concentrations would be 1, 2, 5, 10 and 25 ppbv.

One of the calibration points from the initial calibration curve must be at the same concentration as the daily calibration standard (e.g., 10 ppbv).

10.5.2 Frequency. Each GC/MS system must be recalibrated following corrective action (e.g., ion source cleaning or repair, column replacement, etc.) which may change or affect the initial calibration criteria or if the daily calibration acceptance criteria have not been met.

If time remains in the 24-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

If time does not remain in the 24-hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance check standard followed by analysis of a daily calibration standard.

10.5.3 Procedure. Verify that the GC/MS system meets the instrument performance criteria in Section 10.4.

The GC must be operated using temperature and flow rate parameters equivalent to those in Section 10.2.2. Calibrate the preconcentration-GC/MS system by drawing the standard into the system. Use one of the standards preparation techniques described under Section 9.2 or equivalent.

A minimum of five concentration levels are needed to determine the instrument sensitivity and linearity. One of the calibration levels should be near the detection level for the compounds of interest. The calibration range should be chosen so that linear results are obtained as defined in Sections 10.5.1 and 10.5.5.

Quantitation ions for the target compounds are shown in Table 2. The primary ion should be used unless interferences are present, in which case a secondary ion is used.

## 10.5.4 Calculations.

[Note: In the following calculations, an internal standard approach is used to calculate response factors. The area response used is that of the primary quantitation ion unless otherwise stated.]

10.5.4.1 Relative Response Factor (RRF). Calculate the relative response factors for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

RRF = Relative response factor.

 $A_x$  = Area of the primary ion for the compound to be measured, counts.

 $A_{is}$  = Area of the primary ion for the internal standard, counts.

C<sub>is</sub> = Concentration of internal standard spiking mixture, ppbv.

 $C_x$  = Concentration of the compound in the calibration standard, ppbv.

[Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same for each analysis.  $C_{is}$  and  $C_{x}$  must be in the same units.]

10.5.4.2 Mean Relative Response Factor. Calculate the mean RRF for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n}$$

where:

 $\overline{RRF}$  = Mean relative response factor.

 $x_i = RRF$  of the compound at concentration i.

n = Number of concentration values, in this case 5.

10.5.4.3 Percent Relative Standard Deviation (%RSD). Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(RRF_i - \overline{RRF})^2}{N - 1}}$$

where:

SD<sub>RRF</sub> = Standard deviation of initial response factors (per compound).

RRF<sub>i</sub> = Relative response factor at a concentration level i.

 $\overline{RRF}$  = Mean of initial relative response factors (per compound).

10.5.4.4 Relative Retention Times (RRT). Calculate the RRTs for each target compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{is}}$$

where:

 $RT_c$  = Retention time of the target compound, seconds

 $RT_{is}$  = Retention time of the internal standard, seconds.

10.5.4.5 Mean of the Relative Retention Times ( $\overline{RRT}$ ). Calculate the mean of the relative retention times ( $\overline{RRT}$ ) for each analyte target compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n}$$

where:

RRT = Mean relative retention time for the target compound for each initial calibration standard.

RRT = Relative retention time for the target compound at each calibration level.

10.5.4.6 Tabulate Primary Ion Area Response (Y) for Internal Standard. Tabulate the area response (Y) of the primary ions (see Table 2) and the corresponding concentration for each compound and internal standard.

10.5.4.7 Mean Area Response  $(\overline{Y})$  for Internal Standard. Calculate the mean area response  $(\overline{Y})$  for each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where:

 $\overline{Y}$  = Mean area response.

Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

10.5.4.8 Mean Retention Times  $(\overline{RT})$ . Calculate the mean of the retention times  $(\overline{RT})$  for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where:

 $\overline{RT}$  = Mean retention time, seconds

RT = Retention time for the internal standard for each initial calibration standard, seconds.

10.5.5 Technical Acceptance Criteria for the Initial Calibration.

10.5.5.1 The calculated %RSD for the RRF for each compound in the calibration table must be less than 30% with at most two exceptions up to a limit of 40%.

[Note: This exception may not be acceptable for all projects. Many projects may have a specific target list of compounds which would require the lower limit for all compounds.]

- 10.5.5.2 The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound.
- 10.5.5.3 The area response Y of at each calibration level must be within 40% of the mean area response  $\overline{Y}$  over the initial calibration range for each internal standard.
- 10.5.5.4 The retention time shift for each of the internal standards at each calibration level must be within 20 s of the mean retention time over the initial calibration range for each internal standard.

10.5.6 Corrective Action.

- 10.5.6.1 Criteria. If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the initial calibration technical acceptance criteria.
- 10.5.6.2 Schedule. Initial calibration acceptance criteria <u>must</u> be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed.

## 10.6 Daily Calibration

- 10.6.1 Summary. Prior to the analysis of samples and blanks but after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a daily calibration standard to ensure that the instrument continues to remain under control. The daily calibration standard, which is the nominal 10 ppbv level calibration standard, should contain all the target compounds.
- 10.6.2 Frequency. A check of the calibration curve must be performed once every 24 hours on a GC/MS system that has met the tuning criteria. The daily calibration sequence starts with the injection of the BFB. If the BFB analysis meets the ion abundance criteria for BFB, then a daily calibration standard may be analyzed.
- 10.6.3 Procedure. The mid-level calibration standard (10 ppbv) is analyzed in a GC/MS system that has met the tuning and mass calibration criteria following the same procedure in Section 10.5.
  - 10.6.4 Calculations. Perform the following calculations.

[Note: As indicated earlier, the area response of the primary quantitation ion is used unless otherwise stated.]

- 10.6.4.1 Relative Response Factor (RRF). Calculate a relative response factor (RRF) for each target compound using the equation in Section 10.5.4.1.
- 10.6.4.2 Percent Difference (%D). Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

RRF<sub>c</sub> = RRF of the compound in the continuing calibration standard.

 $\overline{RRF}_i$  = Mean RRF of the compound in the most recent initial calibration.

10.6.5 Technical Acceptance Criteria. The daily calibration standard must be analyzed at the concentration level and frequency described in this Section 10.6 and on a GC/MS system meeting the BFB instrument performance check criteria (see Section 10.4).

The %D for each target compound in a daily calibration sequence must be within ±30 percent in order to proceed with the analysis of samples and blanks. A control chart showing %D values should be maintained.

10.6.6 Corrective Action. If the daily calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the daily calibration technical acceptance criteria.

Daily calibration acceptance criteria must be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed. If the % D criteria are not met, it will be necessary to rerun the daily calibration sample.

# 10.7 Blank Analyses

10.7.1 Summary. To monitor for possible laboratory contamination, laboratory method blanks are analyzed at least once in a 24-hour analytical sequence. All steps in the analytical procedure are performed on the blank

using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

A laboratory method blank (LMB) is an unused, certified canister that has not left the laboratory. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample.

10.7.2 Frequency. The laboratory method blank must be analyzed after the calibration standard(s) and before any samples are analyzed.

Whenever a high concentration sample is encountered (i.e., outside the calibration range), a blank analysis should be performed immediately after the sample is completed to check for carryover effects.

10.7.3 Procedure. Fill a cleaned and evacuated canister with humidified zero air (RH >20 percent, at 25 °C). Pressurize the contents to 2 atm.

The blank sample should be analyzed using the same procedure outlined under Section 10.8.

10.7.4 Calculations. The blanks are analyzed similar to a field sample and the equations in Section 10.5.4 apply.

10.7.5 Technical Acceptance Criteria. A blank canister should be analyzed daily.

The area response for each internal standard (IS) in the blank must be within  $\pm 40$  percent of the mean area response of the IS in the most recent valid calibration.

The retention time for each of the internal standards must be within  $\pm 0.33$  minutes between the blank and the most recent valid calibration.

The blank should not contain any target analyte at a concentration greater than its quantitation level (three times the MDL as defined in Section 11.2) and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

10.7.6 Corrective Action. If the blanks do not meet the technical acceptance criteria, the analyst should consider the analytical system to be out of control. It is the responsibility of the analyst to ensure that contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds.

If an analyte in the blank is found to be out of control (i.e., contaminated) and the analyte is also found in associated samples, those sample results should be "flagged" as possibly contaminated.

#### 10.8 Sample Analysis

10.8.1 Summary. An aliquot of the air sample from a canister (e.g., 500 mL) is preconcentrated and analyzed by GC/MS under conditions stated in Sections 10.1 and 10.2. If using the multisorbent/dry purge approach, adjust the dry purge volume to reduce water effects in the analytical system to manageable levels.

[Note: The analyst should be aware that pressurized samples of high humidity samples will contain condensed water. As a result, the humidity of the sample released from the canister during analysis will vary

in humidity, being lower at the higher canister pressures and increasing in humidity as the canister pressures decreases. Storage integrity of water soluble compounds may also be affected.]

10.8.2 Frequency. If time remains in the 24-hour period in which an initial calibration is performed, samples may be analyzed without analysis of a daily calibration standard.

If time does not remain in the 24-hour period since the injection of the instrument performance check standard in which an initial calibration is performed, both the instrument performance check standard and the daily calibration standard should be analyzed before sample analysis may begin.

- 10.8.3 Procedure for Instrumental Analysis. Perform the following procedure for analysis.
  - 10.8.3.1 All canister samples should be at temperature equilibrium with the laboratory.
  - 10.8.3.2 Check and adjust the mass flow controllers to provide correct flow rates for the system.
- 10.8.3.3 Connect the sample canister to the inlet of the GC/MS analytical system, as shown in Figure 15 [Figure 16 shows an alternate two stage concentrator using multisorbent traps followed by a trap cooled by a closed cycle cooler (15)]. The desired sample flow is established through the six-port chromatographic valve and the preconcentrator to the downstream flow controller. The absolute volume of sample being pulled through the trap must be consistent from run to run.
- 10.8.3.4 Heat/cool the GC oven and cryogenic or adsorbent trap to their set points. Assuming a six-port value is being used, as soon as the trap reaches its lower set point, the six-port chromatographic valve is cycled to the trap position to begin sample collection. Utilize the sample collection time which has been optimized by the analyst.
- 10.8.3.5 Use the arrangement shown in Figure 13, (i.e., a gastight syringe or some alternate method) introduce an internal standard during the sample collection period. Add sufficient internal standard equivalent to 10 ppbv in the sample. For example, a 0.5 mL volume of a mixture of internal standard compounds, each at 10 ppmv concentration, added to a sample volume of 500 mL, will result in 10 ppbv of each internal standard in the sample.
- 10.8.3.6 After the sample and internal standards are preconcentrated on the trap, the GC sampling valve is cycled to the inject position and the trap is swept with helium and heated. Assuming a focusing trap is being used, the trapped analytes are thermally desorbed onto a focusing trap and then onto the head of the capillary column and are separated on the column using the GC oven temperature program. The canister valve is closed and the canister is disconnected from the mass flow controller and capped. The trap is maintained at elevated temperature until the beginning of the next analysis.
- 10.8.3.7 Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning also allows identification of unknown compounds in the sample through searching of library spectra.
- 10.8.3.8 Each analytical run must be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound.
- 10.8.3.9 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the laboratory record book.
  - **10.8.4 Calculations.** The equation below is used for calculating concentrations.

$$C_{x} = \frac{A_{x}C_{is}DF}{A_{is}\overline{RRF}}$$

where:  $C_x = Compound concentration, ppbv.$ 

A<sub>x</sub> = Area of the characteristic ion for the compound to be measured, counts.

 $A_{is}$  = Area of the characteristic ion for the specific internal standard, counts.

C<sub>is</sub> = Concentration of the internal standard spiking mixture, ppbv

 $\overline{RRF}$  = Mean relative response factor from the initial calibration.

DF = Dilution factor calculated as described in section 2. If no dilution is performed, DF = 1

[Note: The equation above is valid under the condition that the volume (~500  $\mu$ L) of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume (~500 mL) of field and QC sample introduced into the trap is the same for each analysis.]

## 10.8.5 Technical Acceptance Criteria.

[Note: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.]

- 10.8.5.1 The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in Sections 10.4, 10.5 and 10.6.
- 10.8.5.2 The field samples must be analyzed along with a laboratory method blank that met the blank technical acceptance criteria.
  - 10.8.5.3 All of the target analyte peaks should be within the initial calibration range.
- 10.8.5.4 The retention time for each internal standard must be within  $\pm 0.33$  minutes of the retention time of the internal standard in the most recent valid calibration.
- 10.8.6 Corrective Action. If the on-column concentration of any compound in any sample exceeds the initial calibration range, an aliquot of the original sample must be diluted and reanalyzed. Guidance inperforming dilutions and exceptions to this requirement are given below.
  - Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
  - The dilution factor chosen should keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

[Note: Analysis involving dilution should be reported with a dilution factor and nature of the dilution gas.]

- 10.8.6.1 Internal standard responses and retention times must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 sec from the latest daily (24-hour) calibration standard (or mean retention time over the initial calibration range), the GC/MS system must be inspected for malfunctions, and corrections made as required.
- 10.8.6.2 If the area response for any internal standard changes by more than  $\pm 40$  percent between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and

corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

10.8.6.3 If, after reanalysis, the area responses or the RTs for all internal standards are inside the control limits, then the problem with the first analysis is considered to have been within the control of the Laboratory. Therefore, submit only data from the analysis with SICPs within the limits. This is considered the initial analysis and should be reported as such on all data deliverables.

## 11. Requirements for Demonstrating Method Acceptability for VOC Analysis from Canisters

#### 11.1 Introduction

- 11.1.1 There are three performance criteria which must be met for a system to qualify under Compendium Method TO-15. These criteria are: the method detection limit of  $\leq 0.5$  ppbv, replicate precision within 25 percent, and audit accuracy within 30 percent for concentrations normally expected in contaminated ambient air (0.5 to 25 ppbv).
- 11.1.2 Either SIM or SCAN modes of operation can be used to achieve these criteria, and the choice of mode will depend on the number of target compounds, the decision of whether or not to determine tentatively identified compounds along with other VOCs on the target list, as well as on the analytical system characteristics.
- 11.1.3 Specific criteria for each Title III compound on the target compound list must be met by the analytical system. These criteria were established by examining summary data from EPA's Toxics Air Monitoring System Network and the Urban Air Toxics Monitoring Program network. Details for the determination of each of the criteria follow.

#### 11.2 Method Detection Limit

- 11.2.1 The procedure chosen to define the method detection limit is that given in the *Code of Federal Regulations* (40 CFR 136 Appendix B).
- 11.2.2 The method detection limit is defined for each system by making seven replicate measurements of the compound of interest at a concentration near (within a factor of five) the expected detection limit, computing the standard deviation for the seven replicate concentrations, and multiplying this value by 3.14 (i.e., the Student's t value for 99 percent confidence for seven values). Employing this approach, the detection limits given in Table 4 were obtained for some of the VOCs of interest.

#### 11.3 Replicate Precision

11.3.1 The measure of replicate precision used for this program is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

percent difference = 
$$\frac{|x_1 - x_2|}{\overline{x}} \times 100$$

where:

 $x_1 =$ First measurement value.

 $x_2$  = Second measurement value.

 $\bar{x}$  = Average of the two values.

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11.3.2 There are several factors which may affect the precision of the measurement. The nature of the compound of interest itself such as molecular weight, water solubility, polarizability, etc., each have some effect on the precision, for a given sampling and analytical system. For example, styrene, which is classified as a polar VOC, generally shows slightly poorer precision than the bulk of nonpolar VOCs. A primary influence on precision is the concentration level of the compound of interest in the sample, i.e., the precision degrades as the concentration approaches the detection limit. A conservative measure was obtained from replicate analysis of "real world" canister samples from the TAMS and UATMP networks. These data are summarized in Table 5 and suggest that a replicate precision value of 25 percent can be achieved for each of the target compounds.

# 11.4 Audit Accuracy

11.4.1 A measure of analytical accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage, as illustrated in the following equation:

Audit Accuracy, 
$$\% = \frac{\text{Spiked Value - Observed Value}}{\text{Spiked Value}} \times 100$$

11.4.2 Audit accuracy results for TAMS and UATMP analyses are summarized in Table 6 and were used to form the basis for a selection of 30 percent as the performance criterion for audit accuracy.

#### 12. References

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#### APPENDIX A.

# LISTING OF SOME COMMERCIAL WATER MANAGEMENT SYSTEMS USED WITH AUTOGC SYSTEMS

Tekmar Dohrman Company 7143 East Kemper Road Post Office Box 429576 Cincinnati, Ohio 45242-9576 (513) 247-7000 (513) 247-7050 (Fax) (800) 543-4461 [Moisture control module]

Entech Laboratory Automation 950 Enchanted Way No. 101 Simi Valley, California 93065 (805) 527-5939 (805) 527-5687 (Fax) [Microscale Purge and Trap]

Dynatherm Analytical Instruments Post Office Box 159 Kelton, Pennsylvania 19346 (215) 869-8702 (215) 869-3885 (Fax) [Thermal Desorption System] XonTech Inc. 6862 Hayenhurst Avenue Van Nuys, CA 91406 (818) 787-7380 (818) 787-4275 (Fax) [Multi-adsorbent trap/dry purge]

Graseby
500 Technology Ct.
Smyrna, Georgia 30082
(770) 319-9999
(770) 319-0336 (Fax)
(800) 241-6898
[Controlled Desorption Trap]

Varian Chromatography System 2700 Mitchell Drive Walnut Creek, California 94898 (510) 945-2196 (510) 945-2335 (FAX) [Variable Temperature Adsorption Trap]

#### APPENDIX B.

#### COMMENT ON CANISTER CLEANING PROCEDURES

The canister cleaning procedures given in Section 8.4 require that canister pressure be reduced to <0.05mm Hg before the cleaning process is complete. Depending on the vacuum system design (diameter of connecting tubing, valve restrictions, etc.) and the placement of the vacuum gauge, the achievement of this value may take several hours. In any case, the pressure gauge should be placed near the canisters to determine pressure. The objective of requiring a low pressure evacuation during canister cleaning is to reduce contaminants. If canisters can be routinely certified (<0.2 ppbv for target compounds) while using a higher vacuum, then this criteria can be relaxed. However, the ultimate vacuum achieved during cleaning should always be <0.2mm Hg.

Canister cleaning as described in Section 8.4 and illustrated in Figure 10 requires components with special features. The vacuum gauge shown in Figure 10 must be capable of measuring 0.05mm Hg with less than a 20% error. The vacuum pump used for evacuating the canister must be noncontaminating while being capable of achieving the 0.05 mm Hg vacuum as monitored near the canisters. Thermoelectric vacuum gauges and turbomolecular drag pumps are typically being used for these two components.

An alternate to achieving the canister certification requirement of <0.2 ppbv for all target compounds is the criteria used in Compendium Method TO-12 that the total carbon count be <10ppbC. This check is less expensive and typically more exacting than the current certification requirement and can be used if proven to be equivalent to the original requirement. This equivalency must be established by comparing the total nonmethane organic carbon (TNMOC) expressed in ppbC to the requirement that individual target compounds be <0.2 ppbv for a series of analytical runs.

## APPENDIX C.

# LISTING OF COMMERCIAL MANUFACTURERS AND RE-SUPPLIERS OF SPECIALLY-PREPARED CANISTERS

BRC/Rasmussen 17010 NW Skyline Blvd. Portland, Oregon 97321 (503) 621-1435

Meriter 1790 Potrero Drive San Jose, CA 95124 (408) 265-6482

Restek Corporation 110 Benner Circle Bellefonte, PA 16823-8812 (814) 353-1300 (800) 356-1688

Scientific Instrumentation Specialists P.O. Box 8941 815 Courtney Street Moscow, ID 83843 (208) 882-3860

Graseby 500 Technology Ct. Smyrna, Georgia 30082 (404) 319-9999 (800) 241-6898

XonTech Inc. 6862 Hayenhurst Avenue Van Nuys, CA 91406 (818) 787-7380

# APPENDIX D.

# LISTING OF COMMERCIAL SUPPLIERS OF PERMEATION TUBES AND SYSTEMS

Kin-Tek 504 Laurel St. Lamarque, Texas 77568 (409) 938-3627 (800) 326-3627

Vici Metronics, Inc. 2991 Corvin Drive Santa Clara, CA 95051 (408) 737-0550

Analytical Instrument Development, Inc. Rt. 41 and Newark Rd. Avondale, PA 19311 (215) 268-3181

Ecology Board, Inc. 9257 Independence Ave. Chatsworth, CA 91311 (213) 882-6795

Tracor, Inc. 6500 Tracor Land Austin, TX (512) 926-2800

Metronics Associates, Inc. 3201 Porter Drive Standford Industrial Park Palo Alto, CA 94304 (415) 493-5632

TABLE 1. VOLATILE ORGANIC COMPOUNDS ON THE TITLE III CLEAN AIR AMENDMENT LIST--MEMBERSHIP IN COMPENDIUM METHOD TO-14A LIST AND THE SOW-CLP LIST OF VOCs

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Compound #9	CAS No.	BP (°C)	V.p. (mmHg) <sup>l</sup>	$MW^{1}$	TO-14A	CLP-SOW
Methyl chloride (chloromethane); CH3Cl	74-87-3	-23.7	3.8 x 10	50.5	X_	X
Carbonyl sulfide; COS	463-58-1	-50.0	3.7 x 10	60.1		
Vinyl chloride (chloroethene); C2H3Cl	75-01-4	-14.0	3.2 x 10	62.5	X	X
Diazomethane; CH2N2	334-88-3	-23.0	2.8 x 10	42.1	_	
Formaldehyde; CH2O	50-00-0	-19.5	2.7 x 10	30		
1,3-Butadiene; C4H6	106-99-0	-4.5	2.0 x 10	54		X
Methyl bromide (bromomethane); CH3Br	74-83-9	3.6	1.8 x 10	94.9	X	X
Phosgene; CCl2O	75-44-5	8.2	1.2 x 10	99		
Vinyl bromide (bromoethene); C2H3Br	593-60-2	15.8	l.l x 10	107		
Ethylene oxide; C2H4O	75-21-8	10.7	1.1 x 10	44		
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	12.5	1.0 x 10	64.5	X	X
Acetaldehyde (ethanal); C2H4O	<b>75-</b> 07-0	21.0	952	44		
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4	31.7	500	97	X	X
Propylene oxide; C3H6O	75-56-9	34.2	445	58		
Methyl iodide (iodomethane); CH3I	74-88-4	42.4	400	141.9		
Methylene chloride; CH2Cl2	75-09-2	40.0	. 349	84.9	X	X
Methyl isocyanate; C2H3NO	624-83 <b>-</b> 9	59.6	348	57.1		
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	44.5	340	76.5	X	X
Carbon disulfide; CS2	75-15-0	46.5	260	76		
Methyl tert-butyl ether; C5H12O	1634-04 <b>-</b> 4	55.2	249	86		
Propionaldehyde; C2H5CHO	123 <b>-</b> 38-6	49.0	235	58.1		
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75 <b>-</b> 34-3	57.0	230	99	Χ	

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TABLE 1. (continued)

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Compound	CAS No	BP(°C)	v.p. (mmHg) <sup>l</sup>	MW <sup>1</sup>	TO-14A	CLP-SOW
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8	59.4	226	88.5		
Chloromethyl methyl ether; C2H5ClO	107-30-2	59.0	224	80.5		
Acrolein (2-propenal); C3H4O	107-02-8	52.5	220	56	<u> </u>	X
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7	63.0	163	72		
Chloroform; CHCl3	67-66-3	61.2	160	119	X	X
Ethyleneimine (aziridine); C2H5N	151-56-4	56	160.0	43		
1,1-Dimethylhydrazine; C2H8N2	57-14-7	63	157.0	60.0		
Hexane; C6H14	110-54-3	69.0	120	86.2	X	
1,2-Propyleneimine (2-methylaziridine); C3H7N	75-55-8	66.0	112	57.1		
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	77.3	100	53	X	
Methyl chloroform (1,1,1-trichloroethane); C2H3Cl3	71-55 <b>-</b> 6	74.1	100	133.4	Х	X
Methanol; CH4O	67 <b>-</b> 56-1	65.0	92.0	32		X
Carbon tetrachloride; CCl4	56-23-5	76.7	90.0	153.8	X	X
Vinyl acetate; C4H6O2	108-05-4	72.2	83.0	86		Χ
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3	79.6	77.5	72		_X
Benzene; C6H6	71-43-2	80.1	76.0	78	X	X
Acetonitrile (cyanomethane); C2H3N	75-05-8	82	74.0	41.0		X
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	83.5	61.5	99	X	Х
Triethylamine; C6H15N	121-44-8	89.5	54.0	101.2		
Methylhydrazine; CH6N2	60-34-4	87. 8	49.6	46.1		
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5	97.0	42.0	113	X	X
2,2,4-Trimethyl pentane C8H18	540-84-1	99.2	40.6	114		
1,4-Dioxane (1,4-Diethylene oxide); C4H8O2	123-91-1	101	37.0	88		
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1	104	30.0	115		
Ethyl acrylate; C5H8O2	140-88-5	100	29.3	100		
Methyl methacrylate; C5H8O2	80-62-6	101_	28.0	100.1		

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TABLE 1. (continued)

Compound	CAS No:	BP (°C)	v.p. (mmgHg)	, "MW <sup>1</sup>	TO-14A	CLP-SOW
Methyl methacrylate; C5H8O2	80-62-101	. 101	28.0	100.1		
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	112	27.8	111	X	X
Toluene; C7H8	108-88-3	111	22.0	92	X	X
Trichloroethylene; C2HCl3	79-01-6	87.0	20.0	131.4	X	X
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	114	19.0	133.4	X	X
Tetrachloroethylene; C2Cl4	127-18-4	121	14.0	165.8	X	Х
Epichlorohydrin (1-chloro-2,3-epoxy propane); C3H5ClO	106-89-8	117	12.0	92.5		
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	132	11.0	187.9	X	Х
N-Nitroso-N-methylurea; C2H5N3O2	684-93-5	124	10.0	103		
2-Nitropropane; C3H7NO2	79-46 <b>-</b> 9	120	10.0	89		
Chlorobenzene; C6H5Cl	108-90-7	132	8.8	112.6	X	X
Ethylbenzene; C8H10	100-41-4	136	7.0	106	·X	X
Xylenes (isomer & mixtures); C8H10	1330-20-7	142	6.7	106.2	Х	X
Styrene; C8H8	100-42-5	145	6.6	104	Х	X
p-Xylene; C8H10	106-42-3	138	6.5	106.2	Х	X
m-Xylene; C8H10	108-38-3	139	6.0	106.2	X	X
Methyl isobutyl ketone (hexone); C6H12O	108-10-1	117	6.0	100.2		·
Bromoform (tribromomethane); CHBr3	75-25-2	149	5.6	252.8		
l,l,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	146	5.0	167.9	X	X
o-Xylene; C8H10	95-47-6	144	5.0	106.2	Х	Х
Dimethylcarbamyl chloride; C3H6CINO	79-44-7	166	4.9	107.6		
N-Nitrosodimethylamine; C2H6N2O	62-75-9	152	3.7	74		
Beta-Propiolactone; C3H4O2	57-57 <b>-</b> 8	Decomposes at 162	3.4	72	·	
Cumene (isopropylbenzene); C9Hl2	98-82-8	153	3.2	120		

TABLE 1. (continued)

	I ADLE I.	(continuea)				
Compound	CAS No.	BP:(°C)	v p (mmHg)	MW <sup>1</sup>	TO-14A	CLP-SOW
Cumene (isopropylbenzene); C9H12	98-82-8	153	3,2	120		
Acrylic acid; C3H4O2	79-10 <b>-</b> 7	141	3.2	72		
N,N-Dimethylformamide; C3H7NO	68-12-2	153	2.7	73		
1,3-Propane sultone; C3H6O3S	1120-71-4	180/30mm	2.0	122.1		
Acetophenone; C8H8O	98-86-2	202	1.0	120		
Dimethyl sulfate; C2H6O4S	77-78-1	188	1.0	126.1		
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	179	1.0	126.6	Χ .	X
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8	196	0.80	236.4		
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4	178	0.71	143	· · · · · · · · · · · · · · · · · · ·	
Chloroacetic acid; C2H3ClO2	79-11-8	189	0.69	94.5		
Aniline (aminobenzene); C6H7N	62-53 <b>-</b> 3	184	0.67	93		
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	173	0.60	147	X.	X
Ethyl carbamate (urethane); C3H7NO2	51-79-6	183 <sup>.</sup>	0.54	89		
Acrylamide; C3H5NO	79-06-1	125/25 mm	0.53	71		
N,N-Dimethylaniline; C8H11N	121-69-7	192	0.50	121		
Hexachloroethane; C2Cl6	67 <b>-</b> 72-1	Sublimes at 186	0.40	236.7		
Hexachlorobutadiene; C4Cl6	87-68-3	215	0.40	260.8	X	X
Isophorone; C9H14O	78-59-1	215	0.38	138.2		
N-Nitrosomorpholine; C4H8N2O2	59-89-2	225	0.32	116.1		
Styrene oxide; C8H8O	96-09-3	194	0.30	120.2		
Diethyl sulfate; C4H10O4S	64-67-5	208	0.29	154		
Cresylic acid (cresol isomer mixture);C7H8O	1319-77-3	202	0.26	108		
o-Cresol; C7H8O	95-48-7	191	0.24	108		
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94		

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TABLE 1. (continued)

Compound:	CAS No.	BP((°C))	v.p. (mmHg) <sup>l</sup>	$MW^1$	TO-14A	CLP-SOW
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94	·	ļ.
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	213	0.18	181.5	Х	Х
nitrohenzene C6H5NO2	98-95-3	211	0.15	123		

<sup>1</sup>Vapor pressure (v.p.), boiling point (BP) and molecularweight (MW) data from:
(a)D. L. Jones and J. bursey, "Simultaneous Control of PM-10 and Hazardous Air Pollutants II: Rationale for Selection of Hazardous Air Pollutants as Potential Particulate Matter," Report EPA-452/R-93/013, U. S. Environmental Protection Agency, Research Triangle Park, NC. October 1992;
(b)R. C. Weber, P. A. Parker, and M. Bowser. Vapor Pressure Distribution of Selected Organic Chemicals, Report EPA-600/2-81-021, U. S. Environmental Protection Agency, Cincinnati, OH, February 1981; and (c)R. C. Weast, ed., "CRC Handbook of Chemistry and Physics," 59th edition, CRC Press, Boca Raton, 1979.

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TABLE 2. CHARACTERISTIC MASSES (M/Z) USED FOR QUANTIFYING THE TITLE III CLEAN AIR ACT AMENDMENT COMPOUNDS

Compound			Secondary Ion
Methyl chloride (chloromethane); CH3Cl	74-87-3	50	52
Carbonyl sulfide; COS	463-S8-1	60	62
Vinyl chloride (chloroethene); C2H3Cl	7S-01-4	62	64
Diazomethane; CH2N2	334-88-3	42 .	41
Formaldehyde; CH2O	50-00-0	29	30
1,3-Butadiene; C4H6	106-99-0	39	54
Methyl bromide (bromomethane); CH3Br	74-83-9	94	96
Phosgene; CCl2O	75-44-5	63	65
Vinyl bromide (bromoethene); C2H3Br	593-60-2	106	108
Ethylene oxide; C2H4O	75-21-8	29	44
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	64	66
Acetaldehyde (ethanal); C2H4O	75-07-0	44	29, 43
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4	61	96
Propylene oxide; C3H6O	75-56-9	58	57
Methyl iodide (iodomethane); CH3I	74-88-4	142	127
Methylene chloride; CH2Cl2	75-09-2	49	84, 86
Methyl isocyanate; C2H3NO	624-83-9	57	56
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	76	41, 78
Carbon disulfide; CS2	75-15-0	76	44, 78
Methyl tert-butyl ether; C5H12O	1634-04-4	73	41, 53
Propionaldehyde; C2H5CHO	123-38-6	58	29, 57
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3	_ 63	65, 27
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8	88	53, 90
Chloromethyl methyl ether; C2H5ClO	107-30-2	45	29, 49
Acrolein (2-propenal); C3H4O	107-02-8	56	55
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7	42	41, 72
Chloroform; CHCl3	67-66-3	83	85, 47
Ethyleneimine (aziridine); C2H5N	151-56-4	42	43
1,1-Dimethylhydrazine; C2H8N2	57-14-7	60	45, 59
Hexane; C6H14	110-54-3	57	41, 43
1,2-Propyleneimine (2-methylazindine); C3H7N	75-55-8	56	57, 42
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	53	52
Methyl chloroform (1,1,1 trichloroethane); C2H3Cl3	71-55-6	97	99, 61
Methanol; CH4O	67-56-1	31	29
Carbon tetrachloride; CCl4	56-23-5	117	119
Vinyl acetate; C4H6O2	108-05-4	43	86
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3	43	72

TABLE 2. (continued)

TABLE 2. (continued	1)		
Compound	CAS No.	Primary Ion	Secondary Ion
Benzene; C6H6	71-43-2	78	77,50
Acetonitrile (cyanomethane); C2H3N	75-05-8	41	40
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	62	64, 27
Triethylamine; C6H15N	121-44-8	86	58, 101
Methylhydrazine; CH6N2	60-34-4	46	31, 45
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5	63	41,62
2,2,4-Trimethyl pentane; C8H18	540-84-1	57	41,56
1,4-Dioxane (1,4 Diethylene oxide); C4H8O2	123-91-1	88	58
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1	79	49, 81
Ethyl acrylate; C5H8O2	140-88-5	55	73
Methyl methacrylate; C5H8O2	80-62-6	41	69, 100
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	75	39, 77
Toluene; C7H8	108-88-3	91	92
Trichloethylene; C2HCl3	79-01-6	130	132, 95
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	97	83, 61
Tetrachloroethylene; C2Cl4	127-18-4	166	164, 131
Epichlorohydrin (1-chloro-2,3-epoxy propane); C3H5ClO	106-89-8	57	49, 62
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	107	109:
N-Nitrso-N-methylurea; C2H5N3O2	684-93-5	60	44, 103
2-Nitropropane; C3H7NO2	79-46-9	43	41 -
Chlorobenzene; C6H5Cl	108-90-7	112	77, 114
Ethylbenzene; C8H10	100-41-4	91	106
Xylenes (isomer & mixtures); C8H10	1330-20-7	91	106
Styrene; C8H8	100-42-5	104	78, 103
p-Xylene; C8H10	106-42-3	91	106
m-Xylene; C8H10	108-38-3	91	106
Methyl isobutyl ketone (hexone); C6H12O	108-10-1	43	58, 100
Bromoform (tribromomethane); CHBr3	75-25-2	173	171, 175
1,1,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	83	85
o-Xylene; C8H10	95-47-6	91	106
Dimethylcarbamyl chloride; C3H6ClNO	79-44-7	72	107
N-Nitrosodimethylamine; C2H6N2O	62-75-9	74	42
Beta-Propiolactone; C3H4O2	57-57-8	42	43
Cumene (isopropylbenzene); C9H12	98-82-8	105	120
Acrylic acid; C3H4O2	79-10-7	72	45, 55
N,N-Dimethylformamide; C3H7NO	68-12-2	73	42, 44
1,3-Propane sultone; C3H6O3S	1120-71-4	58	65, 122

TABLE 2. (continued)

Method TO-15 VOCs

Compound	CAS No.	Primary Ion	Secondary Ion
Acetophenone; C8H8O	98-86-2	105	77,120
Dimethyl sulfate; C2H6O4S	77-78-1	95	66,96
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	91	126
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8	57	155, 157
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4	93	63, 95
Chloroacetic acid; C2H3ClO2	79-11-8	50	45, 60
Aniline (aminobenzene); C6H7N	62-53-3	93	66
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	146	148, 111
Ethyl carbamate (urethane); C3H7NO2	51-79-6	31	44, 62
Acrylamide; C3H5NO	79-06-1	44	55, 71
N,N-Dimethylaniline; C8H11N	121-69-7	120	77, 121
Hexachloroethane; C2Cl6	67-72-1	201	199, 203
Hexachlorobutadiene; C4Cl6	87-68-3	225	227, 223
Isophorone; C9H14O	78-59-1	82	138
N-Nitrosomorpholine; C4H8N2O2	59-89-2	56	86, 116
Styrene oxide; C8H8O	96-09-3	91	120
Diethyl sulfate; C4H10O4S	64-67-5	45	59, 139
Cresylic acid (cresol isomer mixture); C7H8O	1319-77-3	<u> </u>	<u></u>
o-Cresol; C7H8O	95-48-7	108	107
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	110	64
Phenol; C6H6O	108-95-2	94	66
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	180	182, 184
Nitrobenzene; C6H5NO2	98-95-3	77	51, 123

TABLE 3. REQUIRED BFB KEY IONS AND ION ABUNDANCE CRITERIA

ION ABUNDANCE CRITERIA						
Mass Ion Abundance Criteria 1						
50	8.0 to 40.0 Percent of m/e 95					
75	30.0 to 66.0 Percent of m/e 95					
95	Base Peak, 100 Percent Relative Abundance					
96	5.0 to 9.0 Percent of m/e 95 (See note)					
173	Less than 2.0 Percent of m/e 174					
174	50.0 to 120.0 Percent of m/e 95					
175	4.0 to 9.0 Percent of m/e 174					
176	93.0 to 101.0 Percent of m/e 174					
177	5.0 to 9.0 Percent of m/e 176					

 $<sup>^{1}</sup>$ All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 4 METHOD DETECTION LIMITS (MDL)1

TABLE 4. METHOD DET	Lab #1, ŠČAN	Lab #2, SIM
Benzene	0.34	0.29
Benzyl Chloride	_	_
Carbon tetrachloride	0.42	0.15
Chlorobenzene	0.34	0.02
Chloroform	0.25	0.07
1,3-Dichlorobenzene	0.36	0.07
1,2-Dibromoethane		0.05
1,4-Dichlorobenzene	0.70	0.12
1,2-Dichlorobenzene	0.44	_
1,1-Dichloroethane	0.27	0.05
1,2-Dichloroethane	0.24	_
1,1-Dichloroethene	_	0.22
cis-1,2-Dichloroethene	`-	0.06
Methylene chloride	1.38	0.84
1,2-Dichloropropane	0.21	_
cis-1,3-Dichloropropene	0.36	
trans-1,3-Dichloropropene	0.22	_
Ethylbenzene	0.27	0.05
Chloroethane	0.19	
Trichlorofluoromethane		_
1,1,2-Trichloro-1,2,2-trifluoroethane		_
1,2-Dichloro-1,1,2,2-tetrafluoroethane		
Dichlorodifluoromethane	-	
Hexachlorobutadiene		-
Bromomethane	0.53	_
Chloromethane	0.40	
Styrene	1.64	0.06
1,1,2,2-Tetrachloroethane	0.28	0.09
Tetrachloroethene	0.75	0.10
Toluene	0.99	0.20
1,2,4-Trichlorobenzene		
1,1,1-Trichloroethane	0.62	0.21
1,1,2-Trichloroethane	0.50	
Trichloroethene	0.45	0.07
1,2,4-Trimethylbenzene		
1,3,5-Trimethylbenzene		_
Vinyl Chloride	0.33	0.48
m,p-Xylene	0.76	0.08
o-Xylene	0.57	0.28

<sup>1</sup>Method Detection Limits (MDLs) are defined as the product of the standard deviation of seven replicate analyses and the student's "t" test value for 99% confidence. For Lab #2, the MDLs represent an average over four studies. MDLs are for MS/SCAN for Lab #1 and for MS/SIM for Lab #2.

TABLE 5. SUMMARY OF EPA DATA ON REPLICATE PRECISION (RP) FROM EPA NETWORK OPERATIONS1

Monitoring Compound	EPA's Url P	ban Air Toxics I rogram (UATM		EPA's Toxics Air Monitoring Stations (TAMS)			
Identification	%RP	#	ppbv	%RP	#	ppbv	
Dichlorodifluoromethane Methylene chloride	16.3	07	4.3	13.9 19.4	47 47	0.9 0.6	
1,2-Dichloroethane 1,1,1-Trichloroethane	36.2 14.1	31 44	1.6 1.0	 10.6	 47	 2.0	
Benzene Trichloroethene	12.3 12.8	56 08	1.6 1.3	4.4	47 	1.5 	
Toluene Tetrachloroethene Chlorobenzene	14.7 36.2 20.3	76 12 21	3.1 0.8 0.9	3.4	47 	3.1 	
Ethylbenzene m-Xylene	14.6 14.7	32 75	0.7 4.0	5.4 5.3	47 47	0.5 1.5	
Styrene o-Xylene	22.8	59²	1.1	8.7 6.0	47 47	0.2 <sup>2</sup> 0.5	
p-Xylene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	49.1 14.7	06 14	0.6 6.5			 	

Denotes the number of replicate or duplicate analysis used to generate the statistic. The replicate precision is defined as the mean ratio of absolute difference to the average value.

2Styrene and o-xylene coelute from the GC column used in UATMP. For the TAMS entries, both values were

TABLE 6. AUDIT ACCURACY (AA) VALUES¹ FOR SELECTED -COMPENDIUM METHOD TO-14A COMPOUNDS

Selected Compounds From TO-14A List	FY-88 TAMS AA(%), N=30	FY-88 UATMP AA(%), N=3
Vinyl chloride	4.6	17.9
Bromomethane		6.4
Trichlorofluoromethane	6.4	
Methylene chloride	8.6	31.4
Chloroform		4.2
1,2-Dichloroethane	6.8	11.4
1,1,1-Trichloroethane	18.6	11.3
Benzene	10.3	10.1
Carbon tetrachloride	12.4	9.4
1,2-Dichloropropane		6.2
Trichloroethene	8.8	5.2
Toluene	8.3	12.5
Tetrachloroethene	6.2	<b></b>
Chlorobenzene	10.5	11.7
Ethylbenzene	12.4	12.4
o-Xylene	16.2	21.2

<sup>&</sup>lt;sup>1</sup>Audit accuracy is defined as the relative difference between the audit measurement result and its nominal value divided by the nominal value. N denotes the number of audits averaged to obtain the audit accuracy value. Information is not available for other TO-14A compounds because they were not present in the audit materials.

below detection limits for 18 of 47 replicates and were not included in the calculation.

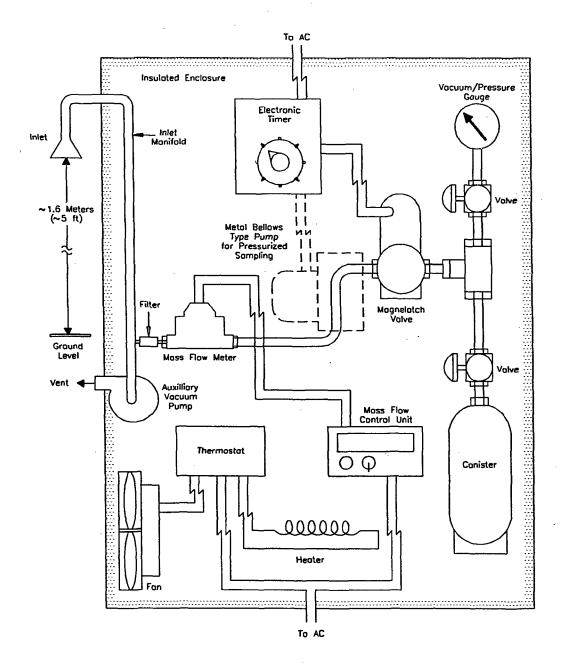
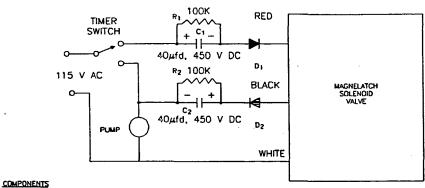
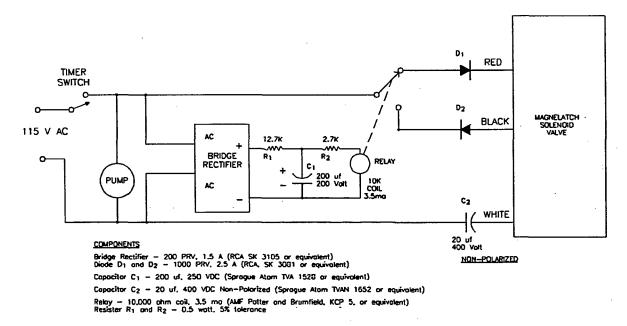


Figure 1. Sampler configuration for subatmospheric pressure or pressurized canister sampling.



Capacitor C1 and C2 - 40 vf. 450 VDC (Sprague Atom. TVA 1712 or equivament) Resister R1 and R2 - 0.5 watt. 5% tolerance. Diode D1 and D2 - 1000 PRV. 2.5 A (RCA, SK 3031 or equivalent)

# (a). Simple Circuit for Operating Magnelatch Valve



(b). Improved Circuit Designed to Handle Power Interruptions

Figure 2. Electrical pulse circuits for driving Skinner magnelatch solenoid valve with mechanical timer.

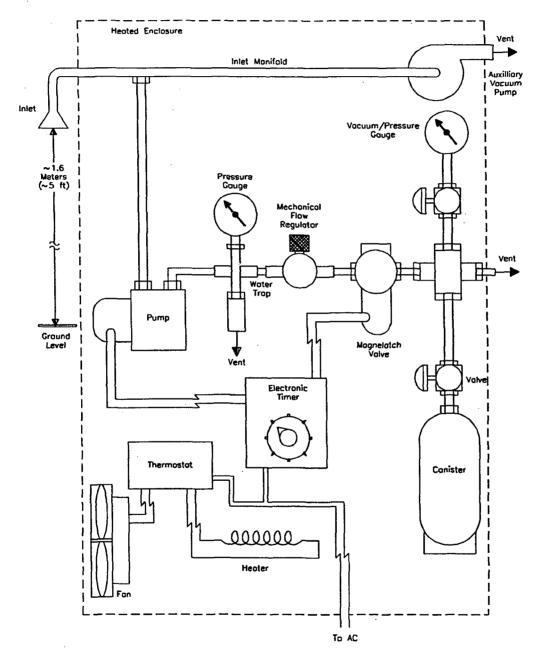


Figure 3. Alternative sampler configuration for pressurized canister sampling.

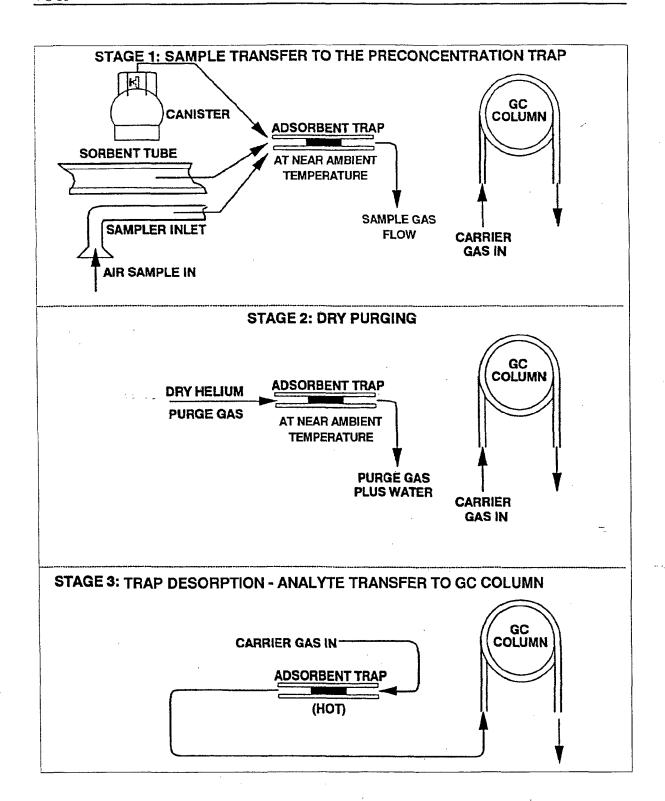


Figure 4. Illustration of three stages of dry purging of adsorbent trap.



Method TO-15

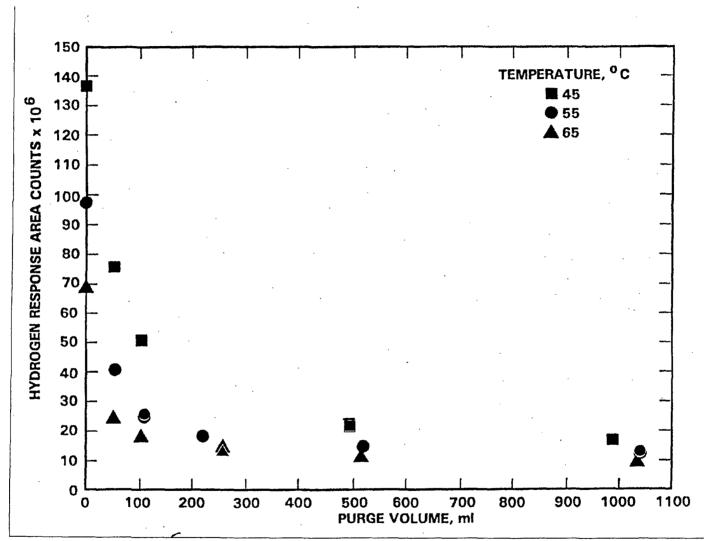


Figure 5. Residual water vapor on VOC concentrator vs. dry He purge volume.

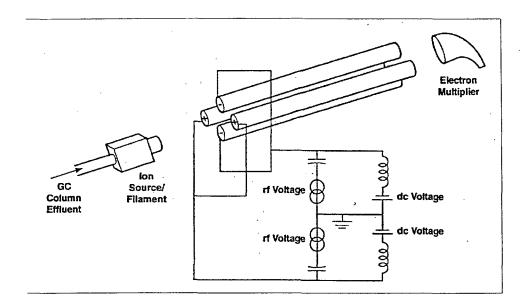


Figure 6. Simplified diagram of a quadrupole mass spectrometer.

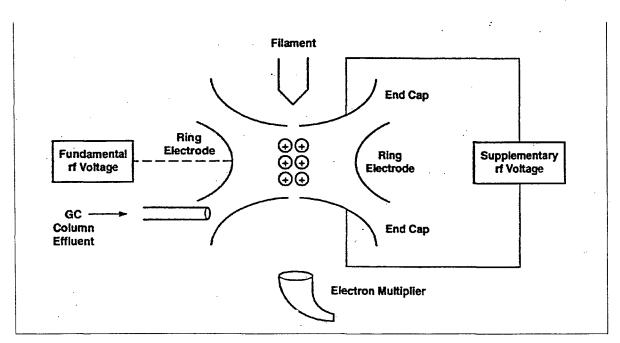


Figure 7. Simplified diagram of an ion trap mass spectrometer.

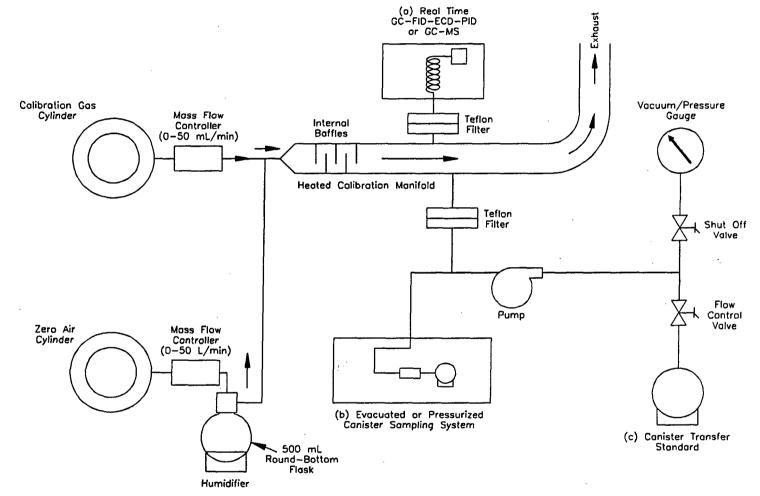


Figure 8. Schematic diagram of calibration system and manifold for (a) analytical system calibration, (b) testing canister sampling system and (c) preparing canister transfer standards.

# COMPENDIUM METHOD TO-15 CANISTER SAMPLING FIELD TEST DATA SHEET

SITE LOCATION:SITE ADDRESS:			<u> </u>	SHIPPING DATE:			
SAMPLING DATE:							
				CANISTER LEA			
				CHECK DAT	TE:		
CAMP	I INC INCODMAC	ION					
SAMP	LING INFORMAT		ED A THE ID IT			DDDCCLIDE	
	D. AMADE TO D	·	ERATURE	Type and	7	PRESSURE	
	INTERIOR	AMBIENT	MAXIMUM	MINIMUM		STER PRESSURE	
TART					<u> </u>	tue discrete	
TOP				<u> </u>			
	SAMPLING TIMES			<del></del>	FLOW RATES		
	LOCAL TIME	ELAPSED T METER REA		MANIFOLD FLOW RATE	CANISTER	FLOW	
		METER REA	DING	FLOW RATE	FLOW RATE	CONTROLLER READOUT	
START	Ţ <u>-</u>						
	<del> </del>	<del> </del>				<del> </del>	
STOP	<u> </u>	<u> </u>			<u> </u>	<u> </u>	
UARTEF	G SYSTEM CERTIF RLY RECERTIFICAT	ΓΙΟΝ DATE:					
UARTEF LABO DATA RECE	RLY RECERTIFICATORY INFORMATION RECEIVED:	TION DATE:					
UARTEF LABO DATA RECE INITIA	RLY RECERTIFICATORY INFORMATION RECEIVED:	TION DATE:		·			
UARTER LABO DATA RECE INITIA FINAL DILUT	RLY RECERTIFICATORY INFORMATION RECEIVED:  IVED BY:  AL PRESSURE:  PRESSURE:  ITION FACTOR:	TION DATE:				:-	
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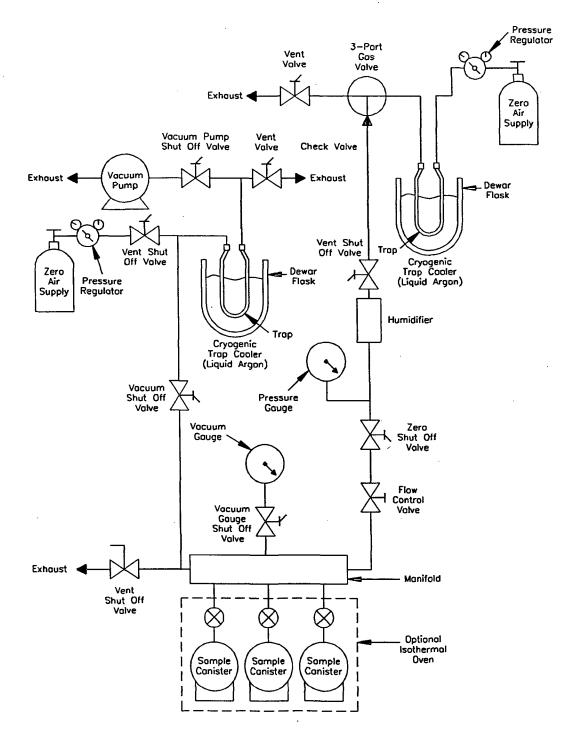


Figure 10. Canister cleaning system.

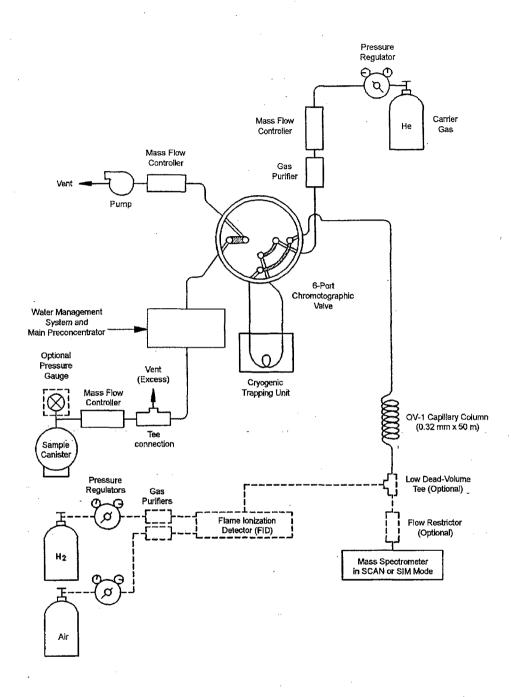
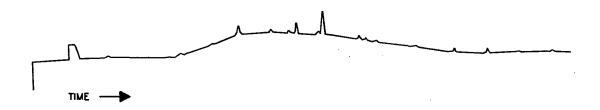
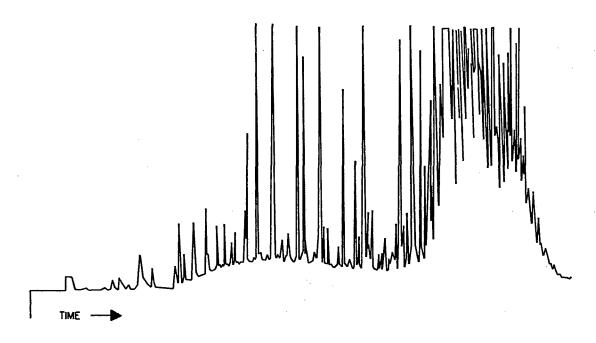


Figure 11. Canister analysis utilizing GC/MS/SCAN/SIM analytical system with optional flame ionization detector with 6-port chromatographic valve in the sample desorption mode.



(a). Certified Sampler



(b). Contaminated Sampler

Figure 12. Example of humid zero air test results for a clean sample canister (a) and a contaminated sample canister (b).

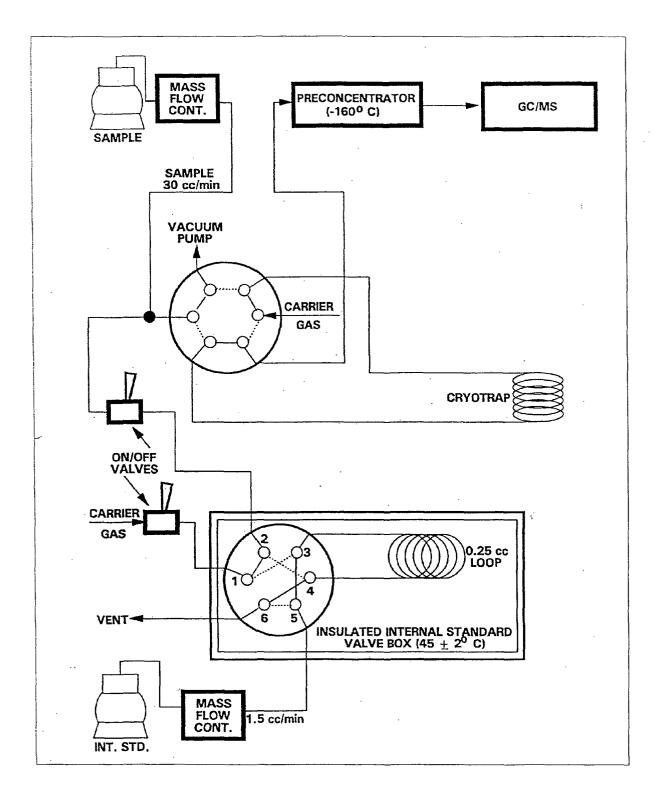


Figure 13. Diagram of design for internal standard addition.

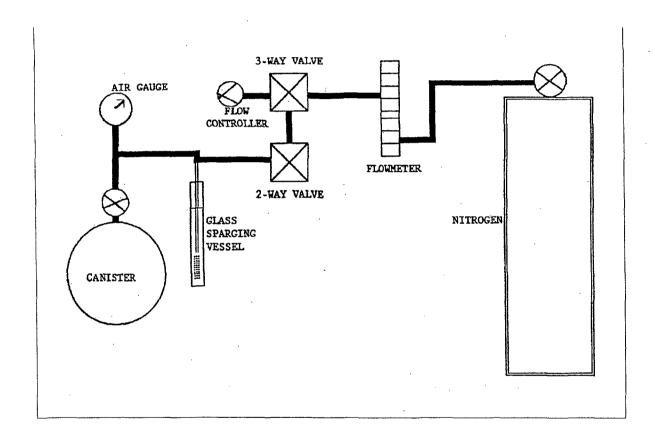


Figure 14. Water method of standard preparation in canisters.

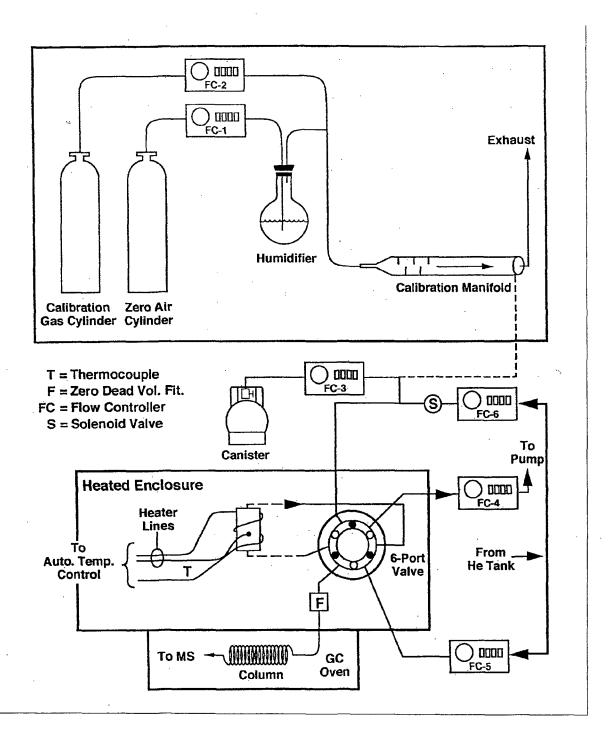


Figure 15. Diagram of the GC/MS analytical system.

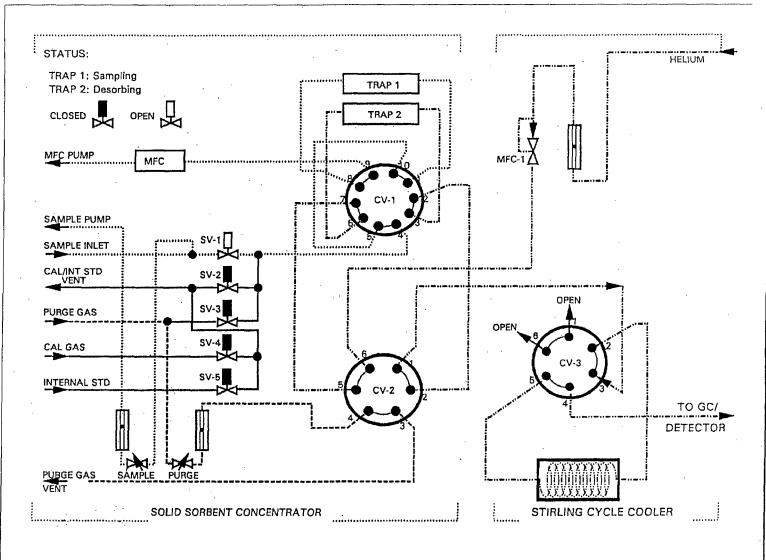


Figure 16. Sample flow diagram of a commercially available concentrator showing the combination of multisorbent tube and cooler (Trap 1 sampling; Trap 2 desorbing).

Attachment 2
Standard Operating Procedures

### CH2M HILL – SOP 1 Collection of Sub-Slab Vapor Samples Using Summa<sup>™</sup> Canisters

#### 1.0 Introduction

This procedure describes the approach for the collection of sub slab soil vapor samples in Summa canisters. It includes instruction on probe installation, leak checking, gas sampling, and probe abandonment. This SOP should be used in conjunction with project data quality objectives. Only persons trained in the collection of soil gas samples should attempt this procedure

#### 2.0 Materials

#### 2.1 Probe Installation

- Hammer drill and 7/8" or 1" and 5/16" or 3/8" bits
- Vacuum cleaner ('shop vac' type or hand held)
- Probe (1/4" stainless steel tube with Swagelock or equivalent nut and ferrule)
- Probe seal (1/8" NPT internal wrenching plug)
- Probe union (1/4" male Swagelock or equivalent to 1/8" female NPT)
- Nuts and ferrules (1/4" stainless steel Swagelock or equivalent)
- Hack saw
- Mortar consisting of Portland Cement mix, Fix-it-All, or similar
- Large Q-tips or paper towels and water
- Tongue depressor, putty knife, or similar tool
- Tape measure

#### 2.2 Leak check

- Leak check enclosure
- Compressed helium tank (balloon grade), helium regulator, flow meter (0-500 ml/min)
- Helium detector, or equal
- (alternative) Isopropyl alcohol and paper towels

#### 2.3 Sampling

- Sampling union (1/4" male Swagelock or equivalent to 1/4" male NPT)
- Vacuum pump, sampling manifold
- ¼" Teflon tubing, rubber tubing
- Flow controller
- Summa canister (sized appropriately for sampling requirements)
- Miscellaneous fitting to connect tubing to sampling union and Summa canister)

#### 2.4 Probe Abandonment

- Probe removal fitting
- Crowbar

#### 2.5 Miscellaneous

- Teflon tape
- 9/16", ½", crescent wrench, screw driver
- Extension cord
- Timer/watch
- Tools required to cut carpet, and/or tools needed for removal of other floor coverings

#### 3.0 Probe Installation

- Locate the sampling locations in accordance with the work plan. Check with local utility companies
  to identify utilities coming into the building from outside. If possible, look for known or suspected
  utility conduits and note their location on a map or in the field log. Be sure to confirm that the
  sample locations will not interfere with the known underground utilities. Also note the location of
  the probe, locations of significant features (walls, cracks, sumps, drains, etc), and conditions of the
  slab and soil.
- If needed, expose the concrete by cutting the carpet or other loose floor coverings (Note: Carpet need not be removed, but rather a 'L' shape cut to expose the concrete for drilling and the leak check enclosure). Drill a 7/8" or 1" diameter hole to a depth of 1-3/4" (measured to the center of the hole) to allow room for the installation of the probe nut and probe union (See Figure 2). Remove the cuttings using a vacuum cleaner. Be careful to not compromise the integrity of the slab during drilling (i.e., cracking it), although note if this occurs. It is important that the slab and the probe hole remain air tight for sampling and that cracks are noted.
- Drill a 5/16" or 3/8" diameter hole through the remainder of the slab and approx. 3" down into the sub-slab material (See Figure 3). Drilling into the sub slab material creates a void that is free of obstructions that might plug the probe during sampling. Record the total depth of the slab and the depth drilled into the sub slab material.
- Once the total depth of the slab is known, be sure that the probe (1/4" stainless steel tube with Swagelock or equivalent nut and ferrule) is cut with a hacksaw so that it does not extend beyond the bottom of the slab. Attach the probe union (1/4" male Swagelock to 1/8" female NPT) to the probe and tighten. Wrap the probe seal with Teflon tape and tighten into the union. See Figure 8 for an expanded view of the probe parts.
- Wet the walls of the hole using the Q-tip or moistened paper towel. This helps the mortar bond to the drilled concrete. Prepare the mortar in accordance with manufacturer's directions to a stiff consistency. Make sure that the consistency is such that the mixture will not run down the sides of the hole and potentially clog the probe or hole. Place sample probe and sample union part way into the hole as shown in Figure 4. The probe tip should be at least one inch into the smaller diameter hole, but not so far that mortar can not be easily placed in the large diameter hole around the probe fittings. It is critical that the mortar mixture does not get into either end of the probe and cause a plug. Using the tongue depressor or similar tool, apply mortar around the base of the sampling probe and sampling union such that it will be sealed once it is in place.
- Fill the hole with mortar, and press the probe further into the hole until its top is flush with the floor. In doing so, slightly wiggle the probe to create good 'wetting' contact between the probe and the mortar as well as the mortar and the drilled concrete. Scrape off excess and make sure there is clear access to the probe. See Figure 5.
- · Let dry for 24 hours
- Be sure to never leave the probe hole open to atmosphere for extended periods to minimize the
  effects of surface infiltration.

#### 3.1 Manifold Assembly and leak check

- Make sure the sampling system is assembled (as shown in Figure 1 or 10) by connecting the sampling
  manifold to the soil gas probe and the purge system. Do not connect the flow controller or canister at
  this time.
- Make sure the gas probe valve (valve #1) is closed.
- Open the sample valve (valve #2) and the purge valve (valve #3) and turn the vacuum pump on. Make sure that the flow meter on the vacuum pump exhaust is reading 200 ml/min. Let the pump run for 1 minute to allow purging of potential contaminants from the manifold
- Now close valve #2 to achieve a vacuum gauge reading of 10 inches of mercury or to a vacuum that will be encountered during sampling, which ever is greater. Close the purge valve #3 and shut the vacuum pump off.

- If the pressure in the manifold has not changed after a minute, then the manifold is considered leak free. If not, repair any leaks prior to use and re-check the manifold.
- Record the leak check date and time on the field sampling log.

#### 3.2 Probe Leak Check and Probe Purging

• The sampling system needs to be leak-checked and purged before sampling. Two different methods supported by the available literature/guidance are presented. One method uses helium gas as a tracer and allows for the assessment of potential probe leak prior to sample collection. The other method uses isopropyl alcohol as a tracer and allows for the assessment of potential probe leak after sample collection and analysis. Both these methods are acceptable and are detailed below.

#### 3.2.1 Helium leak checking technique and purge

- Remove the probe seal insert, wrap the sampling union fitting threads (1/8" male NPT to ¼" male Swagelock or equivalent) with Teflon tape (wrap the NPT threads only), and tighten into the exposed probe fitting. Thread the Teflon sample tubing through the rubber grommet in the leak check enclosure from the outside, and attach the tube to the sampling union using a nut and ferrule. Slide the enclosure down so it seals on the concrete slab. Attach the other end of the sample tube to the sampling manifold. See Figure 6 and 7.
- Attach tubing to the flow meter on the helium tank regulator and the other end to the enclosure. Attach the exhaust tube to the enclosure and position the other end as far away as possible to avoid detection by the helium leak detector. See figure 9.
- Put the helium detector on the exhaust line from the sample pump. Make sure valve 1 is closed. Open valves 2 and 3. Turn on the sample pump and helium detector.
- Open the helium tank and set the flow meter for approximately 200 ml/min. Allow it to flow for 1 minute to fill the leak check enclosure before starting the purge. Make sure that the detector is not reading any helium before starting the purge.
- Two liters of sub slab gas need to be purged before sampling. The purge time is 10 minutes at a flow rate of 200 ml/min. Close valve #2 and open valve #1 simultaneously and start timing for the purge volume. During the purge, observe the helium detector for indication of probe leakage (e.g. infiltration of room air into the probe). If a reading of >5% is observed, then the probe leak check has failed, and corrective action is required see step 4.2.2.
- At the end of the purge time, close valves #1 and #3 and turn the pump off. If at any time during the
  purge the detector read < 5%, then the system is leak free and ready for sampling. If >5% was
  observed, then check the fittings and try again. If <5% cannot be achieved, then this probe must be
  abandoned and a new hole drilled. Be sure to record the helium leak check value on the field sheet.</li>
- Close the helium tank valve.

### 3.2.2 Isopropyl alcohol leak checking technique and purge

- Moisten a paper towel with isopropyl alcohol. Wrap the isopropyl alcohol moistened paper towel around probe fittings at the slab surface. Note: It is important to keep the isopropyl alcohol completely away from the sample equipment and SUMMA Canister during the set up phase. The sampler must also change nitrile gloves between setting up the sample equipment and conducting the leak detection test. It is also important to instruct the laboratory to analyze for isopropyl alcohol. If isopropyl alcohol is detected by the laboratory at a concentration greater than 5% (50,000 ppmv) then the sample is deemed to be invalid due to a leak.
- Two liters of sub slab gas need to be purged before sampling. The purge time is 10 minutes at a flow rate of 200 ml/min. Close valve #2 and open valve #1 simultaneously and start timing for the purge volume.
- At the end of the purge time, close valves #1 and #3 and turn the pump off.

#### 4.0 Sampling

- The Summa canister has been evacuated to near absolute zero pressure. Care should be used at all
  times to prevent inadvertent loss of canister vacuum. Never open the valve affixed to the canister
  unless the intent is to collect sample or check the pressure. Use only a canister that has been certified
  to be clean.
- Remove the canister valve cap, attach the vacuum gauge to the canister, and open the canister valve.
   Record the pressure reading and close and remove the valve. The pressure in the canister should be between 28" and 30" of mercury. If not, then the canister has leaked and should not be used for sampling.
- Connect the flow controller to the manifold (at valve #2) and the Summa canister to the flow controller. The port on the flow controller that reads 'HP' or 'In' should be attached to the manifold. The port that reads 'LP' or 'Out' should be attached to the canister. Use only a flow controller that has been cleaned and properly adjusted.
- To take the sample, <u>confirm valve #3 is closed</u>, and open valves #1 and #2. Slowly open the canister valve approximately one (1) full turn, and start timing. Record the start time on the field sampling log. During the sampling period, record the lowest pressure from the manifold gauge on the field log.
- There are different sample time and flow rate protocols. These are subject to project requirements.
  - o 5 minute sample period, 850 ml canister: The flow controller is set for 150 ml/min. For an 850 cc Summa canister, it will take 5 minutes to collect a sample.
  - o 24 hour sample period, 6 liter canister: The flow controller is set for 3.75 ml/min. For a 6 liter Summa canister, it will take 24 hours to collect a sample.
- At the end of the time, close the sample valve (valve #2) and the valve affixed to the canister. Remove the canister from the apparatus.
- Re-attach the vacuum gauge and record the final pressure. The canister should only have 750 mls of sample in it based on a 150 ml/min sampling rate for 5 minutes. This should equate to between 2" and 5" final pressure in the canister. Record the sampling date, time, canister ID, flow controller ID, and any other observation pertinent to the sampling event on the field sampling log.
- Remove the gauge from the can and tighten the cap back on with a wrench. Verify that canister valve is closed.
- Fill out all appropriate documentation (sampling forms, sample labels, chain of custody, sample tags, etc.).
- Disconnect the sample tubing from the probe, and remove the sampling union.

#### 5.0 Probe Abandonment

- After sampling, it is critical that the probe either be removed or plugged to prevent the creation of a new pathway for vapor intrusion.
- If the probe is to be used again in the future, wrap the probe seal insert with Teflon tape, and tighten it into the probe opening using a hex key until it is tight and flush with the concrete floor.
- If the probe is to be removed, insert the removal fitting into the probe. Using a crow bar, remove the entire probe assembly. If the probe can not be removed in this manner, then over drill the probe with the drill and 1" bit. Fill the hole with cement mix.

Figure 1 - Sub slab Sampling

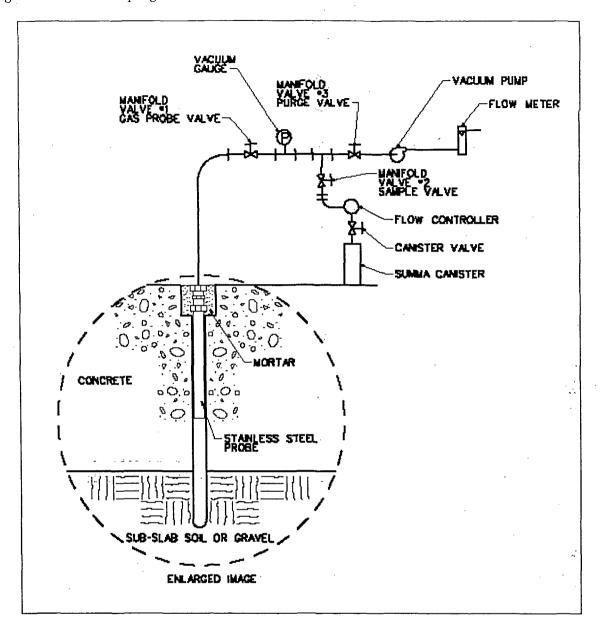


Figure 2 – Drilling 1" mortar hole to a depth of 1 3/4"

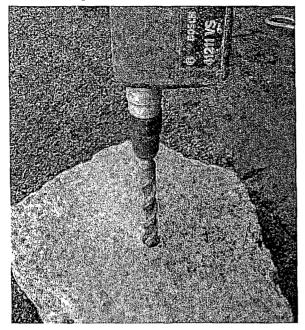


Figure 3 – Drilling 3/8" probe hole

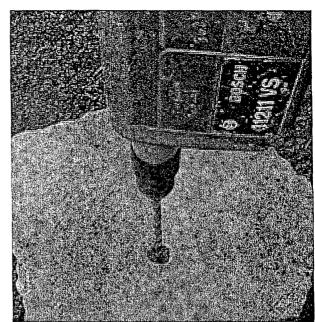


Figure 4 – Installing Probe with mortar

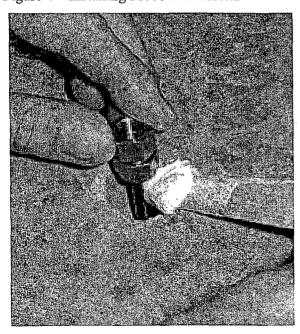


Figure 5 – Installed probe, flush with slab

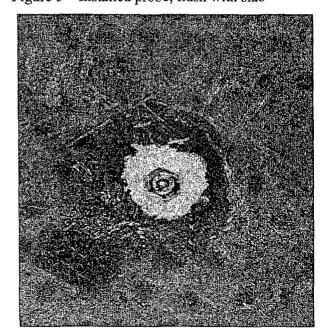


Figure 6 – Installed probe with sample tube

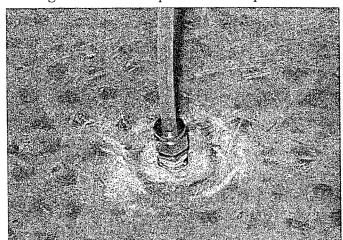


Figure 7 - Installing the helium leak check assembly

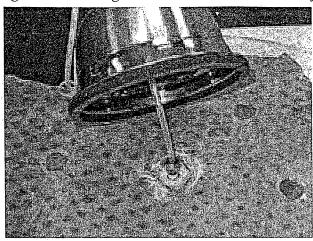


Figure 8 – Probe Parts

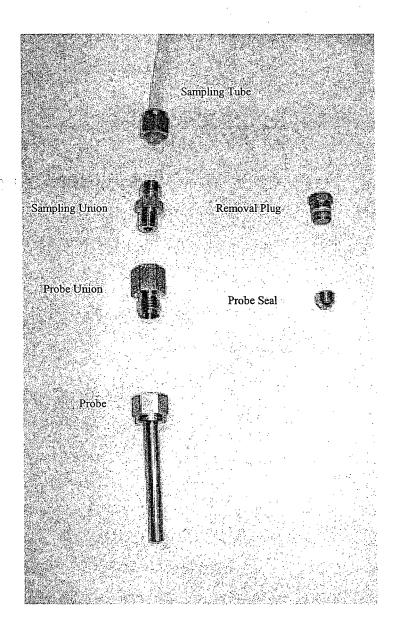


Figure 9 – Helium Leak Check Assembly

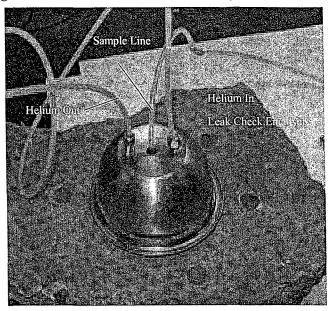
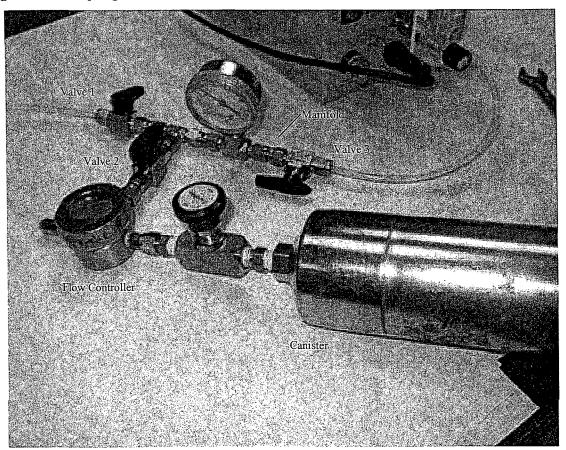


Figure 10 – Sampling Manifold



### CH2M HILL - SOP 2

# TO-14/15 (SUMMA® canister) Integrated Ambient or Indoor Air Sampling Method for Trace VOCs

#### 1.0 Scope and Application

 This sampling method describes the procedure for collecting ambient air samples for targeted volatile organic compounds. The method presented here is based on 'clean' sampling techniques. The requirements of 'clean' sampling dictate sampling and sample handling be by trained personnel.

#### 2.0 Summary of Method

 A sample of ambient air is withdrawn, using clean technique, from into a certified clean and evacuated SUMMA® canister by virtue of a certified clean flow controller. Sample collection can be integrated over time by adjusting the flow controller. Sample periods as short as 10 minutes to as long as 24 hours can be achieved based on the size of canister used and the sampling rate selected.

#### 3.0 Apparatus and Materials

- Canister, SUMMA® polished, certified clean, evacuated, and wrapped for shipping.
- Flow controller, certified clean, set at desired sampling rate, and wrapped for shipping.
- Shipping container, suitable for protection and canister during shipping.
- Wrenches and screw driver (clean and free of contaminants), various sizes as needed for connecting fittings and making adjustment to the flow controller
- Bubble flow meter or equivalent (clean and free of contaminants), used in the adjustment of the flow controller
- Negative Pressure Gauge, oil free and clean, either installed on the canister or used externally to check canister pressure.
- Zero air purge gas (UHP) and regulator to purge and clean flow controller between uses.

#### 4.0 Procedure

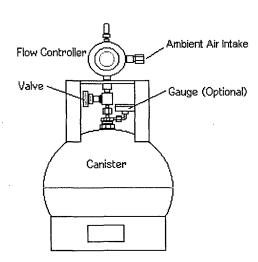
- 'Clean' sampling protocols must be followed at times when handling and collecting samples. This requires care
  in the shipping, storage, and use of sampling equipment. Cleanliness of personnel who come in contact with the
  sampling equipment, no smoking, eating, or drinking, no perfumes, deodorants, clean clothing (not dry cleaned)
  and clean overgarments.
- Inspect the canister for damage. Do not use a canister that has visible damage.
- Verify that the vacuum pressure of the canister is equal to that indicated on the laboratory's evacuation tag. Do
  not use a canister that has leaked.
  - Remove the protective cap from the valve on the canister.
  - o If using an external gauge, attach the gauge to the canister and open the valve. After taking the reading, close the canister and remove the gauge.
- Verify that the flow controller is set at the desired sampling rate. If not pre-set, see the appropriate SOP for flow controller adjustment.
- In the field log record the canister ID, flow controller ID, initial vacuum, desired flowrate, sample location
  information, and all other information pertinent to the sampling effort.
- Connect the flow controller to the canister. The flow controller fitting denoted "LP" or "OUT" is connected to
  the canister. Tighten the fitting as to be leak free but do not over tighten. (A ¼ turn past snug is usually
  enough.)
- Remove all work articles from the sampling area.
- To begin sampling, slowly open the canister valve one full turn.
- For canisters with built in pressure gauges, monitor the vacuum pressure change several times during the course of the selected sample period to ensure the canister is filling at the desired rate.
- At the end of the sample period, close the canister valve finger tight.
- Remove the flow controller and replace the protective cap on the canister valve fitting.

- If using an external vacuum gauge, re-attach it, open the canister valve, and record the pressure. Then close the valve, remove the vacuum gauge, and replace the protective cap.
- If the flow controller is going to be used for more than one sample collection, be sure to purge it between uses.
   To do this, attach the flow controller to a vacuum source and draw clean air or gas (UHP) through it for several minutes before attaching it to the canister.
- Fill out all appropriate documentation (chain of custody, sample tags) and return canisters and all equipment to the laboratory in the shipping containers provided.
- When packing the canisters for shipment, verify that the valve (just past finger tight) and valve caps are snug (1/4 turn past finger tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces.
- Please do not place sticky labels or tape on any surface of the canister!

#### 5.0 Quality Control

- Canister supplied by the laboratory must follow the performance criteria and quality assurance prescribe in US EPA Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. Standard operating procedures are required.
- Flow controllers supplied by the laboratory must follow the performance criteria and quality assurance
  prescribed in US EPA Method TO-14/15 for flow controller cleaning and adjustment. Standard operating
  procedures are required.

FIGURE 1
Assembled Canister Sampler for Integrated Sample Collection



Return to: CH2M HILL

> Applied Sciences Laboratory — Sample Receiving 2300 NW Walnut Boulevard Corvallis, OR 97330-3538

If you have any questions, please call Customer Services at (541) 758-0235, ext. 3120.

### CH2M HILL – SOP 3 TO-14/15 Flow Controller Cleaning (Purging) Method

#### 1.0 Scope and Application

- This method describes the procedure for purge cleaning integrated sampling devices (a.k.a. flow controllers) prior to use in sampling volatile organic compounds (VOCs) from air into evacuated canisters. A flow controller is used to obtain a constant flow rate of an air sample into evacuated canisters over a preset period of time. The flow controller compensates for the decrease in pressure in the canister as it fills up.
- Prior to use, flow controllers must be purged clean of residual VOCs to prevent inadvertent contamination from previous uses. This is accomplished by passing a volume of purge gas through the flow controllers in order to clear any residual VOCs or other contaminants.
- Where the prior use of the flow controller is unknown, the flow controller should be certified clean
  by laboratory analysis. In some cases, such as use in the sampling of process equipment sampling or
  other high VOC sampling applications, purging alone may not be sufficient to clean the flow
  controller.

#### 2.0 Summary of Method

• A flow controller is cleaned by passing a volume of ultra high purity (UHP) air or nitrogen through the controller for a specified amount of time.

#### 3.0 Apparatus and Materials

- Flow controller
- Ultra high purity gas source, air or nitrogen.
- Teflon tubing manifold, with the appropriate fittings for leak-free connection of multiple flow controllers to the as source.
- Extra plugs and caps to plug unused ports.
- Bubble flow meter or equivalent
- Tygon tubing, for leak-free connection of the flow controller to the bubble meter
- Wrenches, various sizes as needed for connecting fittings.

#### 4.0 Procedure

- A schematic of the flow controller purging arrangement is shown in Figure 1.
- Attach the flow controllers to the manifold. Make sure to attach it to the port on the controller that is labeled 'HP' or 'IN'.
- Plug any extra ports in the manifold that are not being used with the stainless steel plugs and caps.
- Attach the source of cleaning gas, and open the valve on the cylinder.
- Check to see that there is flow by attaching the bubble meter to one of the controllers using the 'LP' or 'OUT' port with the Tygon tubing. The other end of the tubing should be attached to the bottom barbed fitting on the bubble meter. The flow should be at least 3 ml/min.
- Repeat this check on several of the controllers.
- Allow the gas to flow for 20-30 minutes. This will allow at least 60 mls of gas to purge the flow controllers.
- Close the purge gas valve.

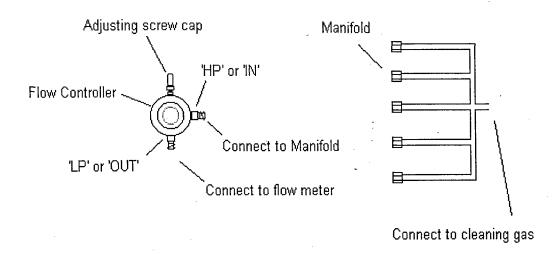
- Disconnect the flow controllers from the manifold.
- Place a suitable seal material (e.g. aluminum foil) over the open ports to prevent dust from getting inside the controller.
- In the field log book or tag provided, record the flow controller ID along with date, time, and who conducted the cleaning.
- The controller's flows are now ready to be checked before the next sampling.

#### 5.0 Quality Control

Flow controllers supplied by the laboratory must follow the performance criteria and quality
assurance prescribed in US EPA Method TO-14/15 for flow controller cleaning and adjustment.
Standard operating procedures are required.

FIGURE 1

Diagram of Flow Controller Cleaning Set Up



Return to: CH2M HILL

Applied Sciences Laboratory — Sample Receiving 2300 NW Walnut Boulevard Corvallis, OR 97330-3538

If you have any questions, please call Ben Thompson at (541) 768-3132, or Mark Boedigheimer at (541) 768-3125.

# CH2M HILL – SOP 4 TO-14/15 Integrated Sampling Device (Flow Controller) Setting Method

#### 1.0 Scope and Application

This method describes the procedure for setting integrated sampling devices (flow controllers) for sampling volatile organic compounds in ambient or indoor air. A flow controller is used to obtain a constant flow rate through the course of sampling an air canister. It compensates for the decrease in pressure in the canister as it fills up.

#### 2.0 Summary of Method

A flow controller is adjusted by measuring the flow of air through the controller, and adjusting the rate until the desired flow is achieved.

#### 3.0 Apparatus and Materials

- Flow controller, pre-cleaned
- Vacuum source, vacuum pump or evacuated can capable of achieving at least 20 inches of vacuum.
- Teflon tubing, with the appropriate fittings for leak-free connection of the flow controller to the vacuum source
- Tygon tubing, for leak-free connection of the flow controller to the bubble meter
- Wrenches and Allen wrench, various sizes as needed for connecting fittings and making adjustment to the flow controller
- Bubble flow meter or equivalent
- Negative Pressure Gauge, to check vacuum source pressure.

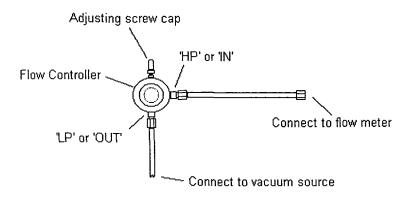
#### 4.0 Procedure

- Attach the flow controller to the vacuum source with the Teflon tube. Make sure to attach it to the port on the controller that is labeled 'LP' or 'OUT'.
- Attach the bubble meter to the controller using the 'HP' or 'IN' port with the Tygon tubing. The other end of the tubing should be attached to the top barbed fitting on the bubble meter.
- Open the vacuum source and take a reading from the flow meter.
- Remove the adjusting screw cap cover from the flow controller.
- Using an Allen wrench, turn the screw clock wise to decrease flow through the flow
  controller, or counter-clockwise to increase the flow. Note: The last turn made must be
  in a clockwise direction. So if the flow needs to be increased, turn the screw counterclockwise a little bit more than necessary, and then turn it clockwise about 1/8 of a turn
  before checking the flow.
- When the desired flow is achieved, replace the adjusting screw cap and take two more readings to insure stability. For a 24 hour sample in a 6 liter can, the flow should be set between 3.5 and 3.75 mls/min.
- Disassemble apparatus.

### 4.0 Quality Control

Flow controllers supplied by the laboratory must follow the performance criteria and quality assurance prescribed in US EPA Method TO-14/15 for flow controller cleaning and adjustment. Standard operating procedures are required.

FIGURE 1
Diagram of Flow Controller



Attachment 3 Example Questionnaire, Field Data Sheet, and Chain of Custody



Industrial Building Data Collection Checklist Potential Vapor Intrusion Pathway Evaluation

Potentiai vap	of mirusion Famway	Evaluatio
Facility:		
	-	ļ
Location:		
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		-

Building Identifier	Building Description				HVAC and Air Infiltration Details											
Room/Building Number or Designation	Room/Building name or description	Floor Space (sq. ft)	Ceiling Height (ft)	Category of Building Use	Estimated total airflow delivered to indoor space (cfm)	Estimated % of air recirculated	Range of Indoor Temperatures (deg F)	Approximate % of wall area as doors or windows	Narrative description of doors/windows (% of time open/closed)							
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Industrial Building Data Collection

Potential Vapor Intrusion Pathway I

Potential Vapo	or Intrusion Pathway I
	,
Facility:	
	·
Location:	

Building Identifier	Building Description	Foundation Details			Comments
				Narrative description of subsurface intrusions (sumps, floor drains,	
Room/Building				basements - including % of floor area	
Number or		Foundation type (i.e. concrete slab,		as basement, extent of cracking in	
Designation	description	wood floor)	Thickness (in)	foundation)	
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Date:	
Preparer;	
Facility:	CH2MHILL
Address:	"Gapt" lid. 30 Jacobs Al. 187 Cl. A. S. S. Sans blog
Contact Person:	
Phone Number:	
e-mail address:	
Building/Room Description	
Building or Room Identifier:	
Primary Activity within Room (select one):	
Manufacturing Storage Other	
Chemical processing Chemical Storage	
Notes:	
	<del></del>
Approximate floor space	
Number of floors	
Multi-room building or Single room	
Ceiling height	
Aboveground Construction Wood Concrete	
Brick Cinderblock	
Other	
Other	
Floor plan attached? Yes No	•
Notes:	

Identification of Site Features	
Physical site features that may be present (check all the	hat apply)
Storage tanks and chemical storage areas Areas with odors present Soil or debris piles Standing pools of liquid Unidentified containers Drains or sumps Stained soils or pavement Degraded floors or walls (i.e. cracks, holes) Pits, ponds, lagoons Dry wells or injection wells Waste treatment, storage or transfer areas Solvent dip or degreasing tanks None of the above present	
Notes:	· · · · · · · · · · · · · · · · · · ·
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Evaluation of Potential Conduits from So	pil .
Floor/foundation description (check all that apply)  Wood Concrete	Elevated above grade?  Below grade?
Other	
Expansion joints present (if concrete floor)?	s No N/A
Are expansion joints sealed?	s No N/A
Are sumps or floor drains present?	es No N/A
Are basements or subsurface vaults present?	es No N/A
Are there subsurface drainage problems?	es No N/A
Notes:	

Evaluation of Potential Pathways/Driving Forces
Are there locations with elevated positive or negative pressure (look for doors not opening/closing properly, perceptible airflow, audible fan noise)
Is there one air conditioning zone or multiple zones (if in a multi-room building)?
Single zone Multi-zone Other
(building management may know; another tip-off is the presence of multiple thermostats = multiple zones)
Sources of outdoor air
Mechanical (air handling unit)  Doors  Windows
Are windows/doors left open routinely?  Yes No
Percent of wall surface area that is doors and/or windows (%)  Total airflow delivered to indoor space (cfm)
Percent airflow recirculated (%)
Notes:
Evaluation of Potential Existing Chemical Sources Indoors
List principal solvent or VOC-containing products used (obtain MSDSs if available)
, and the state of

Description of Vapor	Mitigation Systems	
Has a radon or vapor mitigathis building/room?	ation system been installed in	Yes No
Date of installation?		
Type of system?	Passive venting	Active substab depressurization
	Crack/crevice sealing	Dilution ventilation control
	N/A	
Notes:		
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Date: Preparer:	
Facility:	
Description (floor):	CH2MHIL

#### Floor Plan Information

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Project Name:	·····					Project #:			
Sampling Data Lo	y, the second								
Sample Location	Field ID	Canister ID	Flow Controller ID	Initial Canister Pressure ("Hg)	Initial Flow Controller Rate (ml/min)	Start Date & Time	End Data & Time	Final Pressure ("Hg)	Final Flow Controller Rate (ml/min)
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Sample Location	Diagram								
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	e the structure's found	dation and interior	walls, identify roo	ms, and note	other defining	features. Sh	ow location of	canister relativ	re to
	ts, etc.					TIT			

#### Indoor Vapor Intrusion Assessment Sub-slab Vapor Field Sampling Log - Summa Canister Method

Sheet 1 of 2

	Project Info				
	Project Name:			Project #:	
	Ву:			Date:	
	!Structure * **********************************				
	Identification:				
	Address:				
	Slab Information:  Concrete slab on grade (directly	on top of soil)	Other (describe)		
	Concrete slab on gravel underla				
	Condition of slab				
	Type of Sub Slab Soil				
	Is water present in the soil				
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	Sub-slab Probeilnstallation, Le				
	Samplé location (show in diagram)	1	- 2	3 ·	4
	Sample Identification (field ID)				
Probe Installation	Depth of slab (inches)				. 1
	Depth of hole drilled (inches below slab surface)				
	Depth of installed probe (inches below slab surface)				
Manifold Leak check	Leak check (sampling manifold) - Pass/No Pass				
Probe Purge	Purge rate, cc/min.				
	Purge Start (time of day)				
	Purge vacuum, " Hg		<u></u>		
	Purge completed (time of day)				
Helium Leak Check (optional)	Leak check (Helium) - %				
Canister Sampling	Canister & flow controller ID (if used)				<u>-</u>
	Initial Canister Pressure (* Hg)				
	Sampling rate, cc/min				
	Sampling period started (time of day)				6
	Sampling vacuum, * Hg				<u> </u>
	Sampling period ended (time of day)	1			
	Final Canister Pressure (* Hg)				
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Observations a	and Comments:				
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CVO 2300 NW Walnut Boulevard Corvallis, OR 97330-3538 (541) 752-4271 FAX (541) 752-0276

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### New Jersey Department of Environmental Protection

# INDOOR AIR BUILDING SURVEY and SAMPLING FORM

Preparer's name:	Date:
Preparer's affiliation:	
Site Name:	Case #:
Part I - Occupants	
Building Address:	
Property Contact: Owner /	Renter / other:
Contact's Phone: home ( ) work (	) cell ( )
# of Building occupants: Children under age 13	Children age 13-18 Adults
Part II – Building Characteristics	
Building type: residential / multi-family residential / o	office / strip mall / commercial / industrial
Describe building:	Year constructed:
Sensitive population: day care / nursing home / hospita	l / school / other (specify):
Number of floors below grade: (full basement /	crawl space / slab on grade)
Number of floors at or above grade:	
Depth of basement below grade surface:ft.	Basement size:ft²
Basement floor construction: concrete / dirt / floating	/ stone / other (specify):
Foundation walls: poured concrete / cinder blocks	/ stone / other (specify)
Basement sump present? Yes / No Sump pump? Yes	es / No Water in sump? Yes / No
Type of heating system (circle all that apply):  hot air circulation hot air radiation heat pump hot water radiation other (specify):	wood steam radiation kerosene heater electric baseboard
Type of ventilation system (circle all that apply):  central air conditioning mechanical individual air conditioning units kitchen ran other (specify):	
Type of fuel utilized (circle all that apply):  Natural gas / electric / fuel oil / wood / coal	/ solar / kerosene
Are the basement walls or floor sealed with waterproof pa	aint or epoxy coatings? Yes / No

Is there a whole house fan?	Yes / No
Septic system?	Yes / Yes (but not used) / No
Irrigation/private well?	Yes / Yes (but not used) / No
Type of ground cover outside of build	ling: grass / concrete / asphalt / other (specify)
Existing subsurface depressurization (	(radon) system in place? Yes / No active / passive
Sub-slab vapor/moisture barrier in pla  Type of barrier:	ace? Yes / No
Part III - Outside Contaminant Sou	urces ( )
NJDEP contaminated site (1000-ft. r.	adius):
Other stationary sources nearby (gas	stations, emission stacks, etc.):
Heavy vehicular traffic nearby (or oth	her mobile sources):

#### Part IV – Indoor Contaminant Sources

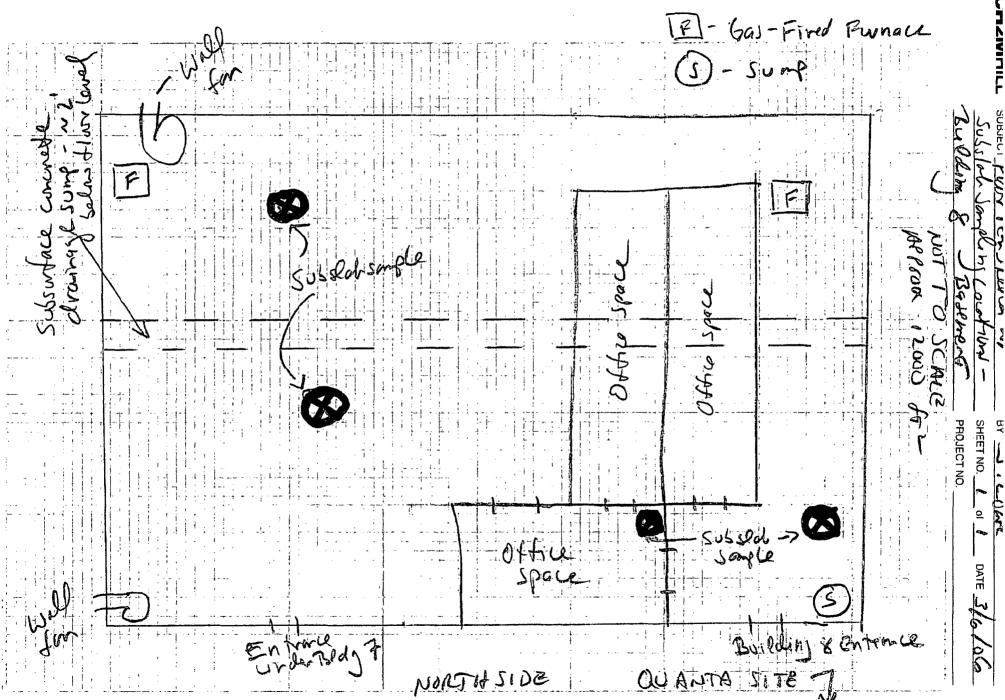
Identify all potential indoor sources found in the building (including attached garages), the location of the source (floor and room), and whether the item was removed from the building 48 hours prior to indoor air sampling event. Any ventilation implemented after removal of the items should be completed at least 24 hours prior to the commencement of the indoor air sampling event.

Potential Sources	Location(s)	Removed (Yes/No/NA)
Gasoline storage cans		(2007.107.112)
Gas-powered equipment		
Kerosene storage cans		
Paints / thinners / strippers		
Cleaning solvents		
Oven cleaners		
Carpet / upholstery cleaners		
Other house cleaning products		
Moth balls		
Polishes / waxes		
Insecticides		
Furniture / floor polish		
Nail polish / polish remover		
Hairspray		
Cologne / perfume		
Air fresheners		
Fuel tank (inside building)		NA NA
Wood stove or fireplace		NA
New furniture / upholstery		
New carpeting / flooring		NA
Hobbies - glues, paints, etc.		

Part V – Miscellaneous Items	
Do any occupants of the building smoke? Yes / No	How often?
Last time someone smoked in the building?	hours / days ago
Does the building have an attached garage directly connected to living	g space? Yes / No
If so, is a car usually parked in the garage? Yes / No	
Are gas-powered equipment or cans of gasoline/fuels stored in	n the garage? Yes / No
Do the occupants of the building have their clothes dry cleaned?	Yes / No
If yes, how often? weekly / monthly / 3-4 times a year	
Do any of the occupants use solvents in work? Yes / No	•
If yes, what types of solvents are used?	
If yes, are their clothes washed at work? Yes / No	
Have any pesticides/herbicides been applied around the building or in	
If so, when and which chemicals?	
Has there ever been a fire in the building? Yes / No	If yes, when?
Has painting or staining been done in the building in the last 6 n	nonths? Yes / No
If yes, when and where?	
Part VI – Sampling Information	
Sample Technician: Phone numb	per: ( )
Sample Source: Indoor Air / Sub-Slab / Near Slab Soil Gas / Exteri	ior Soil Gas
Sampler Type: Tedlar bag / Sorbent / Stainless Steel Canister / Ot	her (specify):
Analytical Method: TO-15 / TO-17 / other: Cer	t. Laboratory:
Sample locations (floor, room):	
Field ID # Field ID #	
	-
Were "Instructions for Occupants" followed?  Yes /No  If not, describe modifications:	

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rt VIII – G	eneral Observation	ons .				(
ovide any in erpretation p	formation that may process.	be pertinent to t	he sampling eve	nt and may assist i	n the data	

Appendix B Floor Plans for 115 River Road Building Basements



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